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CONSTRUCTION PRODUCTIVITY ADVANCEMENT RESEARCH (CPAR) PROGRAM

IMPROVED MATERIALS AND PROCESSES FOR SEALING
AND RESEALING JOINTS IN PORTLAND CEMENT
CONCRETE PAVEMENTS: LABORATORY STUDY

by

Larry N. Lynch, Dewey W. White, James Chehovits

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Construction Productivity and Reduce Costs

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Improved Materials and Processes for Sealing and Resealing Joints in Portland Cement Concrete Pavements: Laboratory Study

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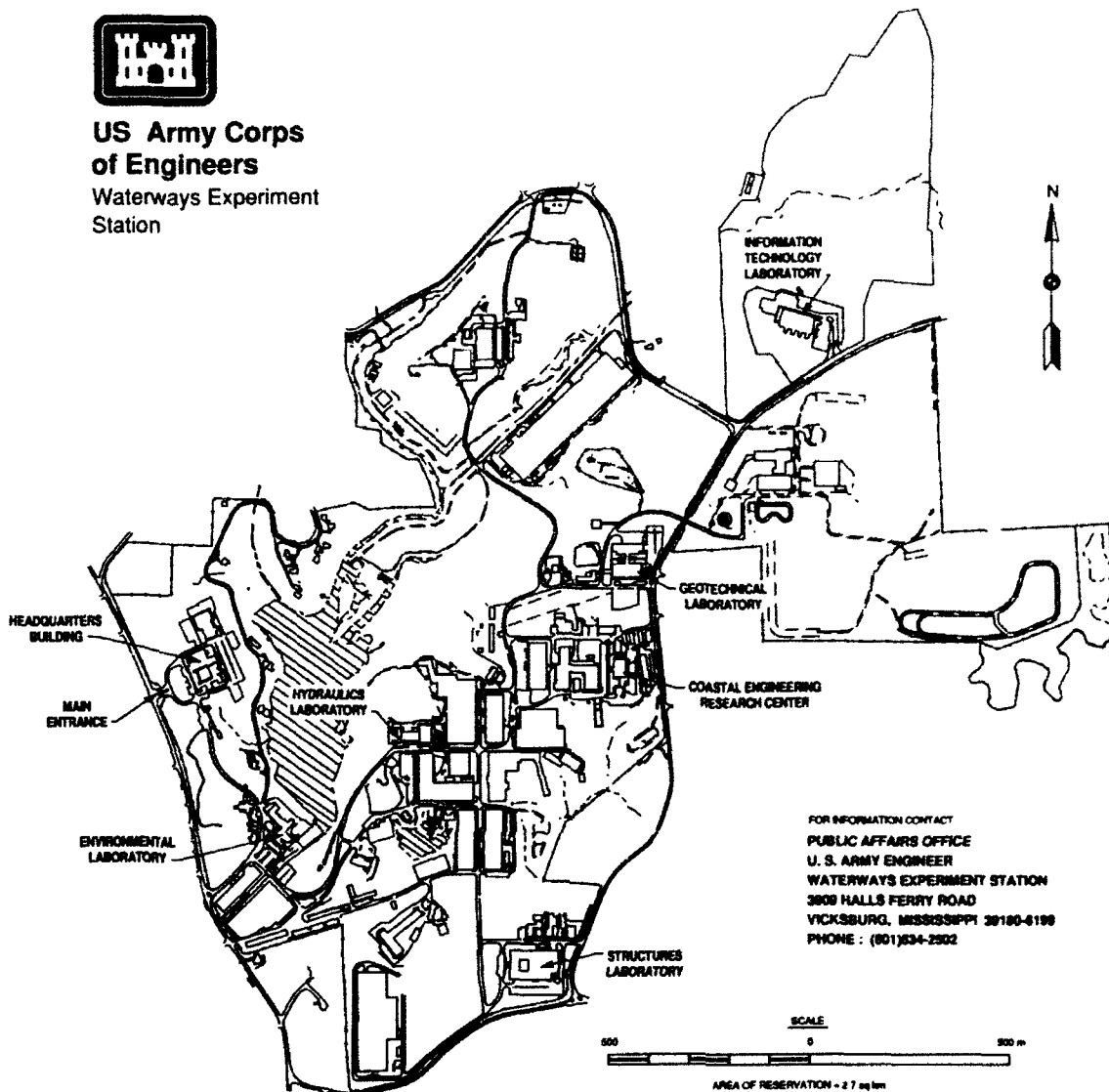
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PREFACE

Section 7 of the Water Resources Development Act of 1988, P.L. 100-676, 33 U.S.C. 2313, and the Stevenson-Wydler Technology Innovation Act of 1980, as amended, 15 U.S.C. 37102a, provide the legislative authority for the Construction Productivity Advancement Research (CPAR) Program. The CPAR program allows the US Army Corps of Engineers to enter into cooperative research and development agreements with construction industry partners to conduct cost-shared, collaborative efforts with the goal of improving construction productivity.

The CPAR project "Improved Materials and Processes for Sealing and Resealing Joints in Portland Cement Concrete Pavements," was a collaborative effort of the Geotechnical Laboratory (GL) of the US Army Engineer Waterways Experiment Station (WES) and Crafcro Incorporated. The work was conducted from October 1989 to December 1991. The US Army Corps of Engineers Technical Monitor was Mr. Gregory Hughes.

The project was conducted under the general supervision of Dr. W. F. Marcuson III, Director, GL, WES, and under the direct supervision of Mr. H. H. Ulery, Jr., former Chief, Pavement Systems Division (PSD); Dr. G. M. Hammitt II, Chief, PSD, GL; Dr. R. S. Rollings, former Chief, Materials Research and Construction Technology (MR&CT), and Mr. T. W. Vollor, Chief, MR&CT. The WES Principal Investigator was Mr. Larry N. Lynch, and the Crafcro Principal Investigator was Mr. James Chehovits. The report was prepared by Messrs. Dewey W. White, Lynch, and Chehovits.

At the time of publication of this report Director of WES was Dr. Robert W. Whalin. Commander was COL Leonard G. Hassell, EN.

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CONVERSION FACTORS, NON-SI TO SI (METRIC)
UNITS OF MEASUREMENT

Non-SI units of measurement used in this report can be converted to SI (metric) units as follows:

<u>Multiply</u>	<u>By</u>	<u>To Obtain</u>
degrees (angle)	0.01745329	radians
Fahrenheit degrees	5/9	Celsius degrees or Kelvins*
inches	2.54	centimetres
ounces (US fluid)	0.02957353	cubic decimetres
pounds (force) per square inch	6.894757	kilopascals
square inches	6.4516	square centimetres

* To obtain Celsius (C) temperature readings from Fahrenheit (F) readings, use the following formula: $C = (5/9)(F - 32)$. To obtain Kelvin (K) readings, use: $K = (5/9)(F - 32) + 273.15$.

IMPROVED MATERIALS AND PROCESSES FOR SEALING AND RESEALING
JOINTS IN PORTLAND CEMENT CONCRETE PAVEMENTS
LABORATORY STUDY

PART I: INTRODUCTION

Background

1. Many types and compositions of sealants are currently used for sealing joints in portland cement concrete (PCC) pavement. These materials vary widely in chemical complexity, field performance, suitability for a specific application, and price. Currently, there are four types of sealants which can be specified on military projects. The specifications for these sealants are:

- a. Federal Specification (FS) SS-S-1401C, "Sealant, Joint, Non-Jet-Fuel Resistant, Hot-Applied, for Portland Cement and Asphalt Concrete Pavements" (FS 1984a).
- b. FS SS-S-1614A, "Sealant, Joint, Jet-Fuel-Resistant, Hot-Applied, for Portland Cement and Tar Concrete Pavements" (FS 1984b).
- c. FS SS-S-200E, "Sealants, Joint, Two Component, Jet-Blast-Resistant, Cold-Applied, for Portland Cement Concrete Pavement" (FS 1988).
- d. US Army Corps of Engineers Handbook for Concrete and Cement CRD-C 527, "Joint Sealants, Cold-Applied, Non-Jet-Fuel-Resistant, for Rigid and Flexible Pavements" US Army Corps of Engineers Handbook of Concrete and Cement 1988).

2. Sealants manufactured to meet the requirements of FS SS-S-1401C are typically polymer modified asphalt-base, hot-applied materials. These sealants are used to seal cracks in asphalt cement pavements, or joints in PCC pavements in areas that are not exposed to fuel spillage. Typical application areas for FS SS-S-1401C sealants are parking lots, roadways, and some portions of taxiways. For personnel not familiar with federal specifications, FS SS-S-1401C is similar, but not identical to American Society for Testing and Materials (ASTM) D3405 (ASTM 1991).

3. Sealants manufactured to meet the requirements of FS SS-S-1614A are polymer modified coal tar-base, hot-applied materials and are used to seal joints in PCC pavements where fuel spillage would be expected. FS SS-S-1614A type sealants should not be used to seal cracks in asphalt pavements. Typical application areas are maintenance areas and aircraft parking aprons. FS SS-S-1614A is somewhat similar to ASTM D3581 (ASTM 1991).

4. Sealants manufactured to meet the requirements of FS SS-S-200E are two-component, cold-applied materials which are typically polysulfide or polyurethane modified tar compositions. These sealants are used to seal joints in PCC pavements where both fuel spillage and aircraft blast are expected. Typical application areas are aircraft warm-up areas, pavements that are exposed to vertical short takeoff landing (VSTOL) aircraft, and the ends of runways. There is not an ASTM specification that is similar to FS SS-S-200E.

5. Sealants manufactured to meet the requirements of CRD-C 527 (US Army Corps of Engineers Handbook of Concrete and Cement 1988) are cold-applied and can be either single or multicomponent materials. CRD-C 527 sealants are used to seal joints and cracks in either PCC or asphalt cement concrete pavements that are not exposed to fuel spillage. Typical areas where these sealants can be used are the same as for FS SS-S-1401C sealants. An ASTM specification that is similar to CRD-C 527 does not exist.

6. Each of the above mentioned pavement joint sealants is manufactured for a specific use; however, an ideal sealant, regardless of the specification to which it was manufactured, would have certain characteristics. These ideal characteristics would include:

- a. Simple and repeatable application techniques.
- b. Adhesion to joint faces when subjected to extreme temperatures and joint movements.
- c. Insensitive to moisture.
- d. Reject intrusion of incompressible materials.
- e. Resistant to long term weathering.
- f. Resistant to various de-icing chemicals.
- g. Resist bubbling and/or blistering during application and service.
- h. Economical.

Special characteristics for particular applications are also desirable. For example, fuel resistance and/or blast resistance are required for some applications.

7. Each sealant type; FS SS-S-1401C, SS-S-1614A, SS-S-200E, and CRD-C527 may possess one more of these ideal characteristics. But no one type of sealant contains all of them.

8. Field observations and evaluations conducted in recent years by various agencies have indicated problem areas associated with the sealing and

resealing of joints in PCC pavements. Some of the specific problems that have been observed include the following:

- a. Some sealants tend to become brittle or hard as atmospheric and pavement temperatures decrease and as sealants age.
- b. Hot-applied sealants have experienced bubbling tendencies during application or within the first few months (usually within 6 months) of application.
- c. Some sealants experience cohesion and/or adhesion failures.

Project Objectives

9. Crafco Incorporated and the US Army Engineer Waterways Experiment Station (WES) conducted a cost-shared collaborative effort to investigate methods of improving pavement joint sealant performance. This program was funded under the auspices of the FY 89 Construction Productivity Advancement Research (CPAR) Program. The stated objectives of the research effort were as follows:

- a. Objective 1: Development of specification limits for a hot-applied, jet-fuel-resistant (JFR) sealant with improved performance characteristics as compared to current FS SS-S-1614A type materials.
- b. Objective 2: Development of specification limits for a hot-applied, non-jet-fuel-resistant (non-JFR) sealant with improved low temperature performance characteristics as compared to current FS SS-S-1401C type materials.
- c. Objective 3: Development of specification limits for a primer system that will minimize bubbling tendencies associated with hot-applied sealants and improve sealant adhesion to PCC.
- d. Objective 4: Develop field data to determine performance of flush fill sealant application geometry versus 1/8 to 1/4 in.* recess application technique.

Purpose of Report

10. Since the project involved both laboratory investigations and field installation information, the project was divided into a laboratory phase, and a field installation phase.

* A table of factors for converting non-SI units of measurement to SI (metric) units is presented on page 5.

11. The purpose of this report is to document the laboratory investigation effort or laboratory phase of the project.

PART II: LABORATORY RESEARCH PLAN

12. The research plans to accomplish the three objectives included in the laboratory phase were very similar. Initial meetings were conducted to identify specific properties and characteristics that would be desirable in an improved hot-applied, JFR sealant, an improved hot-applied, non-JFR sealant, and in a primer system that would minimize the bubbling tendencies of hot-applied sealants. Once the desirable characteristics of the two types of sealants were identified, several JFR and non-JFR sealants were procured.

13. The procured sealants were representative of commercially available non-JFR and JFR sealants. The sealants obtained were not limited to those manufactured to FS SS-S-1401C or FS SS-S-1614A. For example, some of the sealants were manufactured to meet ASTM specifications. More specifically, the non-JFR sealants procured were manufactured to meet the requirements of FS SS-S-1401C, ASTM D3405, or a modified low modulus ASTM D3405 which has been adopted by several state agencies. The procured JFR sealants were manufactured to meet the requirements of FS SS-S-1614A, ASTM D3569, or ASTM D3581. The non-JFR sealants were tested in accordance with FS SS-S-1401C, and the JFR sealants were tested in accordance with FS SS-S-1614A to establish a material properties baseline. Additional testing of each of the sealants was conducted to determine if the hot-applied sealants exhibited the desired characteristics outlined in the initial meetings.

14. Laboratory formulation studies of both non-JFR and JFR sealants were conducted to produce hot-applied sealants which exhibit more of the desired characteristics than those exhibited by commercially available sealants. Based on the laboratory testing of the procured sealants and on the laboratory formulations, draft material specifications were developed to highlight the desired material characteristics. Finally, production quantities of the most promising laboratory formulations were produced for laboratory testing and allow field evaluations.

15. Once the desirable properties and characteristics were defined in the initial meeting, a literature search and laboratory study were initiated. The literature search was conducted to gather information on primer materials used in construction applications. The laboratory study was conducted using the US Army Corps of Engineers Handbook of Concrete and Cement CRD-C 525 (1989) test procedure which evaluates the bubbling tendencies of hot-applied

sealants. The laboratory study was conducted to gain an increased understanding of the sealant bubbling phenomena. Laboratory formulation studies were conducted to develop a non-JFR and a JFR primer material with the identified desirable properties. A draft material specification was developed for the primer system based on the laboratory study and laboratory formulations. Production quantities of primer were produced for testing in accordance with the draft material specification and for future field evaluations.

16. The specific materials, testing procedures, testing results, and development procedures and results are discussed in the remainder of this report.

PART III: PHYSICAL CHARACTERISTICS OF AVAILABLE
HOT-APPLIED, JET-FUEL-RESISTANT SEALANTS

Products Tested

17. During initial project planning meetings, it was determined that prior to initiating development activities for an improved JFR sealant, it would be helpful to test a variety of commercially available hot-applied JFR sealants to determine their physical properties. It was also decided that not only should testing in accordance to FS SS-S-1614A be conducted, but that more extensive testing to determine performance limits and to provide a basis for a comparison and improvement should also be conducted. The sealants submitted were manufactured to meet either FS SS-S-1614A, ASTM D3569, or ASTM D3581.

18. Joint sealants evaluated for the JFR portion of this project were as follows:

- a. Superseal 1614A, Plant 1, Crafco Inc., manufactured to meet the requirements of FS SS-S-1614A and ASTM D3581. This is a liquid material as supplied by the manufacturer.
- b. Superseal 1614A, Plant 2, Crafco Inc., manufactured to meet the requirements of FS SS-S-1614A and ASTM D3581. This is a liquid material as supplied by the manufacturer. The purpose for using two materials from Crafco Inc. was to determine if any differences were evident in products made to meet the same specification, by the same manufacturer, but at different plant locations with different sources of raw materials.
- c. Product 9012, Koch Materials Company, manufactured to meet the requirements of FS SS-S-1614A, ASTM D3581, and ASTM D3569. This is a solid material as supplied by the manufacturer.
- d. NEA-1614, Koch Materials Company, manufactured to meet the requirements of FS SS-S-1614A and ASTM D3581. This is a liquid material as supplied by the manufacturer.
- e. Nitoseal 777, Fosroc International, LTD., manufactured to meet the requirements of ASTM D3569. This is a liquid material as supplied by the manufacturer.

Production lot numbers with the manufacturers' recommended pour and safe heating temperatures are as follows:

<u>Product</u>	<u>Lot Identification</u>	<u>Pour Temperature</u>	<u>Safe Heating Temperature</u>
Superseal 1614A, Plant 1	SACIBO	250°F	270°F
Superseal 1614A, Plant 2	SHM9A9	250°F	270°F
Product 9012	1260	260°F	280°F
NEA-1614	1246	250°F	270°F
Nitoseal 777	3371-T	275°F	300°F

Specification Testing

19. Each of the five sealant materials was tested in accordance with FS SS-S-1614A using the manufacturers' recommended safe heating temperature. FS SS-S-1614A requires the material to be heated for a total of 3 hr before being poured into the sample molds for testing. The sealants were also tested to FS SS-S-1614A required parameters at several other heating periods. The additional heating periods included 45 min to provide an indication of short term heating and application, and 4.5 and 6 hr to provide indications of the effects of heating for longer periods than required by the specification. Selected testing was performed by both WES and CrafcO laboratories to provide an indication of testing variability between laboratories. Summaries of the FS SS-S-1614A testing conducted by CrafcO and WES are presented in Table 1. A data summary which shows mean values, estimated standard deviation (based on a value of 0.886 times the range of the two values) (Burr 1974), and the coefficient of variation is shown in Table 2.

20. CrafcO test results for the JFR sealants tested in accordance with FS SS-S-1614A with heating times of 45 min, 4.5 hr, and 6.0 hr are summarized in Tables 3 through 5.

21. A brief summary of the test procedures is provided in Appendix A.

Specification Test Analysis.

22. The test results provided in Table 1 indicate that the Superseal 1614A from Plant 1, the Product 9012, and the NEA 1614 conformed to the test requirements of FS SS-S-1614A in both the WES and the CrafcO laboratories. The other two sealants, Superseal 1614A from Plant 2 and Nitoseal 777,

Table 1

WES and CrafcO Results for Jet-Fuel-Resistant SealantsTested in Accordance with FS SS-S-1614A

FS SS-S-1614A Test Parameter	<u>Superseal 1614A-1</u>		<u>Superseal 1614A-2</u>		<u>Product 9012</u>		<u>NEA 1614</u>		<u>Nitoseal 777</u>	
	<u>WES</u>	<u>CrafcO</u>	<u>WES</u>	<u>CrafcO</u>	<u>WES</u>	<u>CrafcO</u>	<u>WES</u>	<u>CrafcO</u>	<u>WES</u>	<u>CrafcO</u>
Penetration, cm										
Nonimmersed	1.08	1.22	1.16	1.08	0.96	1.02	0.99	1.08	1.16	1.35
Fuel-immersed	0.89	1.09	0.96	0.89	0.83	0.88	0.84	0.86	0.89	0.95
Change	-0.19	-0.13	-0.20	-0.19	-0.13	-0.14	-0.15	-0.22	-0.27	-0.40
Fuel-immersed										
Weight change, percent	0.20	0.22	0.20	0.19	0.20	0.26	0.20	0.30	0.50	0.50
Flow, cm	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
Bond										
Nonimmersed	S*	S	S	S	S	S	S	S	S	S
Fuel-immersed	S	S	U**	S	S	S	S	S	S	S
Water immersed	S	S	S	S	S	S	S	S	S	S

* S signifies satisfactory performance in accordance with specification requirements.

** U signifies unsatisfactory performance.

Table 2

Summary of FS SS-S-1614A Data Obtained by WES and Crafcro

FS SS-S-1614A Test Parameter	Superseal 1614A, P1*	Superseal 1614A, P**	Product 9012	NEA 1614	Nitoseal 777	Overall Average
Penetration, cm						
Nonimmersed						
\bar{X}	1.15	1.12	0.99	1.04	1.26	1.11
S	0.124	0.071	0.053	0.080	0.168	--
CV	10.8%	6.3%	5.4%	7.7%	13.3%	8.7%
Penetration, cm						
Fuel-immersed						
\bar{X}	0.99	0.925	0.855	0.85	0.92	0.91
S	0.177	0.062	0.044	0.018	0.053	--
CV	17.9%	6.7%	5.2%	2.1%	5.8%	7.5%
Penetration						
Change						
\bar{X}	0.160	0.195	0.135	0.185	0.335	0.202
S	0.053	0.009	0.009	0.062	0.115	--
CV	33.1%	4.6%	6.7%	33.5%	34.3%	22.4%
Fuel-immersed						
Weight change, percent						
\bar{X}	0.210	0.195	0.23	0.25	0.50	0.277
S	0.018	0.009	0.053	0.089	0.0	--
CV	8.6%	4.6%	23.0%	35.6%	0.0%	14.4%
Flow, cm						
\bar{X}	0.00	0.00	0.00	0.00	0.00	0.00
S	0.0	0.0	0.0	0.0	0.0	--
CV	0.0	0.0	0.0	0.0	0.0	0.0

Notes: \bar{X} - mean

S - standard deviation (calculated as 0.886 times the data range)

CV - coefficient of variation

* P1 represents material from Plant 1.

** P2 represents material from Plant 2.

Table 3

Test Results in Accordance with FS SS-S-1614A Using

45 min Heating Period

FS SS-S-1614A Test Parameter	Superseal 1614A, P1*	Superseal 1614A, P2**	Product 9012	NEA 1614	Nitoseal 777	FS SS-S-1614A Requirement
Penetration, cm						
Nonimmersed	1.50	1.23	0.96	1.05	1.40	1.30 max
Fuel-immersed	1.14	0.93	1.00	0.92	0.75	1.55 max
Change	-0.36	-0.30	+0.04	-0.13	-0.65	+0.25 max
Fuel-immersed						
weight change, percent	0.48	0.20	0.25	0.33	0.45	±2.0 max
Flow, cm	0.00	0.00	0.00	0.00	0.00	0.30 max
Bond						
Nonimmersed	S†	S	S	S	S	S
Fuel-immersed	S	S	S	S	S	S
Water immersed	S	S	S	S	S	S

* Material from Plant 1.

** Material from Plant 2.

† S signifies satisfactory performance in accordance with specification requirements.

Table 4

Test Results in Accordance with FS SS-S-1614A Using

4.5 hr Heating Period

FS SS-S-1614A Test Parameter	Superseal 1614A, PI*	Superseal 1614A, P2**	Product 9012	NEA 1614	Nitoseal 777	FS SS-S-1614A Requirement
Penetration, cm						
Nonimmersed	1.14	1.02	0.98	1.13	†	1.30 max
Fuel-immersed	1.01	0.93	0.85	0.87	--	1.55 max
Change	-0.13	-0.09	+0.13	-0.26	--	+0.25 max
Fuel-immersed						
weight change, percent	0.22	0.18	0.28	0.25	--	±2.0 max
Flow, cm	0.00	0.00	0.00	0.00	--	0.30 max
Bond						
Nonimmersed	S††	U†	S	S	--	S
Fuel-immersed	S	S	S	S	--	S
Water immersed	S	U	S	S	--	S

* Material from Plant 1.

** Material from Plant 2.

† Testing was not performed due to insufficient quantity of sealant.

†† S signifies satisfactory performance in accordance with specification requirements.

‡ U signifies unsatisfactory performance.

Table 5

Test Results in Accordance with FS SS-S-1614A Using

6 hr Heating Period

FS SS-S-1614A Test Parameter	Superseal 1614A, P1*	Superseal 1614A, P2**	Product 9012	NEA 1614	Nitoseal 777	FS SS-S-1614A Requirement
Penetration, cm						
Nonimmersed	1.00	0.67	0.95	0.98	1.24	1.30 max
Fuel-immersed	0.89	0.79	0.71	0.81	0.75	1.55 max
Change	-0.11	+0.12	-0.24	-0.17	-0.49	+0.25 max
Fuel-immersed						
weight change, percent	0.10	0.20	0.22	0.25	0.50	±2.0 max
Flow, cm	0.00	0.00	0.00	0.00	0.00	0.30 max
Bond						
Nonimmersed	S†	U††	U	S	S	S
Fuel-immersed	S	U	S	S	S	S
Water immersed	S	U	S	S	S	S

* Material from Plant 1.

** Material from Plant 2.

† S signifies satisfactory performance in accordance with specification requirements.

†† U signifies unsatisfactory performance.

had conflicting reports. The Superseal 1614A from Plant 2 material was reported as conforming to FS SS-S-1614A requirements in the Crafco laboratory tests, but the material exhibited cohesive failure on the second cycle during the testing at WES. The Nitoseal 777 conformed to the test requirements of FS SS-S-1614A at WES, but the material did not meet the nonimmersed penetration requirement as reported by Crafco.

23. The summarized data provided in Table 2 indicate some variability between the testing conducted by WES and Crafco. The nonimmersed penetration average values ranged from 0.99 to 1.26 cm. The range of values indicates the diversity of materials that can meet the FS SS-S-1614A nonimmersed penetration requirement of 1.30 cm maximum. More important than the change in average penetration values is the coefficient of variance (CV) of the nonimmersed penetration results of each of the sealants. The CV ranges from a low of 5.4 percent for the Product 9012 results to a high of 13.3 percent for the Nitoseal 777. The fuel-immersed penetration average values had a narrower range, but the CV ranged from a low of 2.1 percent for the NEA 1614 to a high of 17.9 percent for the Superseal 1614A from Plant 1. The CV for the fuel-immersed change in weight exhibited a larger range than either the non-immersed or fuel-immersed penetration results. The CV ranged from 0.0 percent to 35.6 percent. The flow tests indicated no apparent variability because none of the samples exhibited flow. The simple statistical analysis of the data indicates that variability in the test results does exist between laboratories and that the variability could be of a sufficient magnitude for one laboratory to report that the sealant conforms to the specifications and a second laboratory to report that the sealant does not conform to the specification requirements. The bond testing is reported by satisfactory or unsatisfactory; therefore, statistical analysis is more difficult. The bond test results were very consistent between the laboratories with only one test that did not correspond. The one exception was the fuel-immersed bond testing for the Superseal 1614A from Plant 2. The reason for the discrepancy in the bond testing or the variability of the other test results is not known. However, items such as sampling techniques, melting and heating effects of the laboratory equipment, human variability, and conditions to which the sealants were exposed during shipment could be contributing factors to the noted variances.

24. The data obtained on the sealant materials using the additional heating periods are provided in Tables 3, 4, and 5. Data with numerical

values are plotted in Figures 1 through 4. The data indicate that the physical properties of the sealants change as the heating period is varied.

25. The Superseal 1614A Plant 1 sample did not meet the FS SS-S-1614A test requirements when exposed to the 45 min heating period. The nonimmersed penetration value exceeded the 1.30 cm limit. However, the Superseal 1614A Plant 1 sample did meet the FS SS-S-1614A test requirements when exposed to the longer heating periods of 3.0, 4.5, and 6.0 hr. The longer heating periods appeared to stiffen or harden the Superseal 1614A Plant 1 sample as illustrated by a decrease in both the nonimmersed and fuel-immersed penetration values. The fuel-immersed change in weight also exhibited a decrease with an increase in the heating time, but the flow values and bond to concrete results were not affected.

26. The Superseal 1614A Plant 2 sample met the FS SS-S-1614A test requirements at heating periods of 45 min and 3.0 hr, but did not meet the testing requirements at heating periods of 4.5 and 6.0 hr. The Superseal 1614A sample from Plant 2 hardened as the heating time was extended as indicated by the nonimmersed penetration values. The fuel-immersed penetrations generally decreased with an increase in the heating period. The fuel-immersed penetration value for the 4.5 hr heating period was the exception to the trend. The fuel-immersion change in weight and the flow values did not change appreciably with the increased heating times, but the sealant became sufficiently stiff to result in unsatisfactory bond to concrete for the 4.5 and 6.0 hr heating times.

27. The significance in differing test of the Superseal 1614A Plant 1 and Plant 2 samples is the fact that both materials are manufactured using the same formulations, but from different sources of raw materials. The sealants were affected differently by extended heating, and, therefore, they will probably be affected differently by weathering.

28. Product 9012 met the FS SS-S-1614A testing requirements after the 45 min, 3.0 hr, and 4.5 hr heating periods. However, it did not meet the testing requirements when exposed to the 6.0 hr heating time. The non-immersed penetration and fuel-immersed change-in-weight values did not change appreciably with an increase in the heating period. The fuel-immersed penetration values indicated a stiffening with increased heating times. The flow and bond to concrete results did not change with the exception of the non-immersed bond results at 6.0 hr. The nonimmersed bond test results were

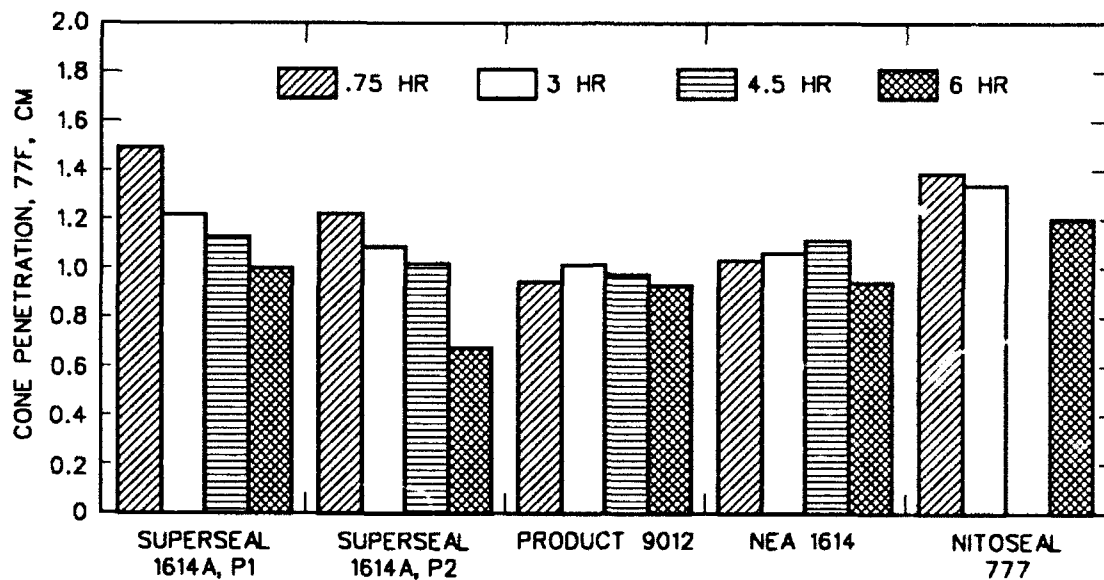


Figure 1. Nonimmersed penetration of jet-fuel-resistant sealants after 0.75, 3, 4.5, and 6 hr of heating

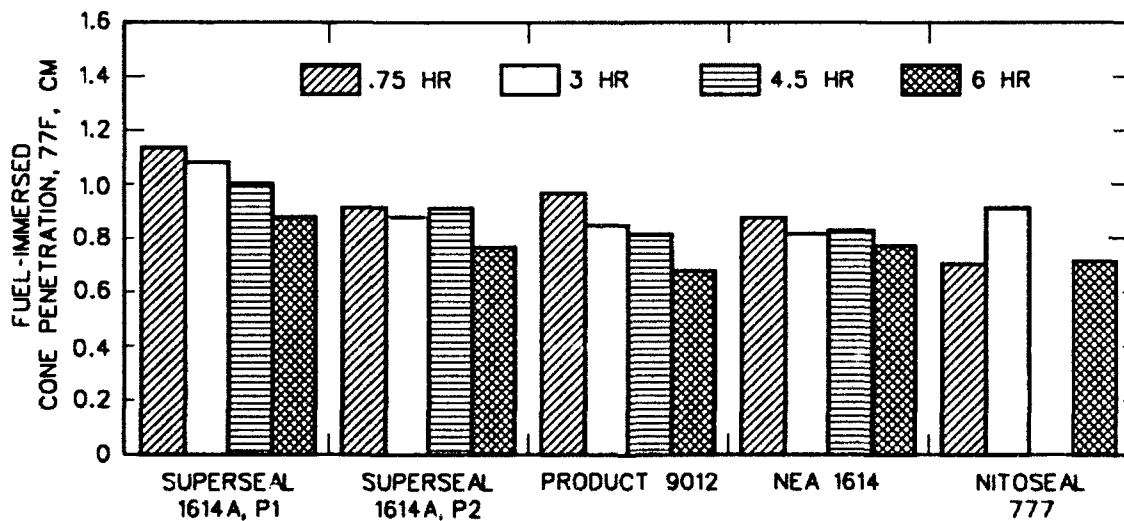


Figure 2. Fuel-immersed penetration of jet-fuel-resistant sealants after 0.75, 3, 4.5, and 6 hr of heating

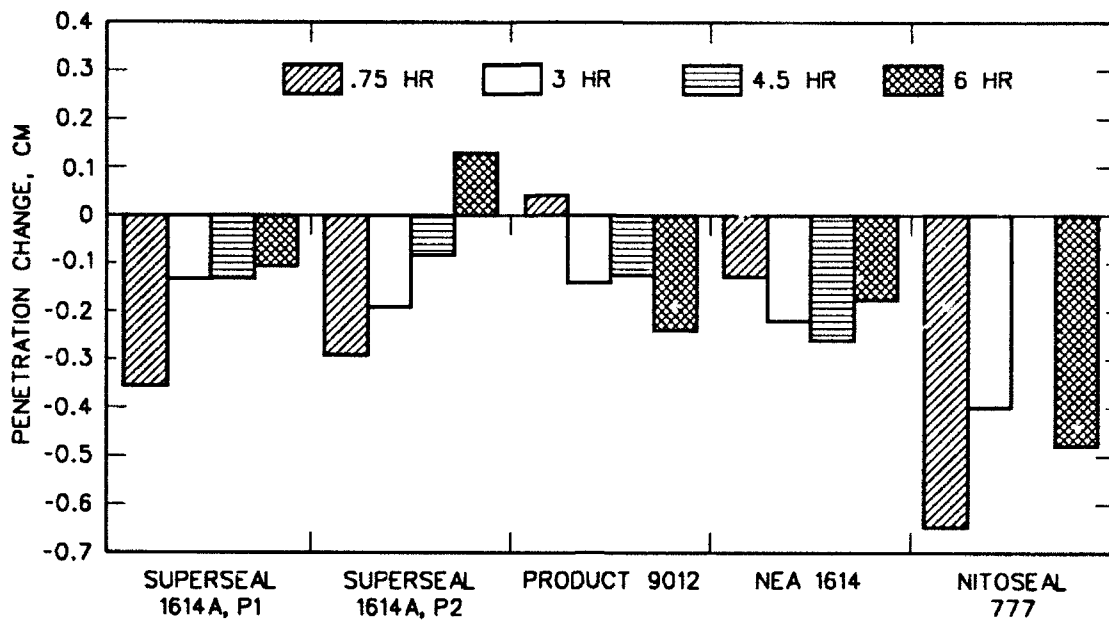


Figure 3. Penetration change of jet-fuel-resistant sealants after 0.75, 3, 4.5, and 6 hr of heating

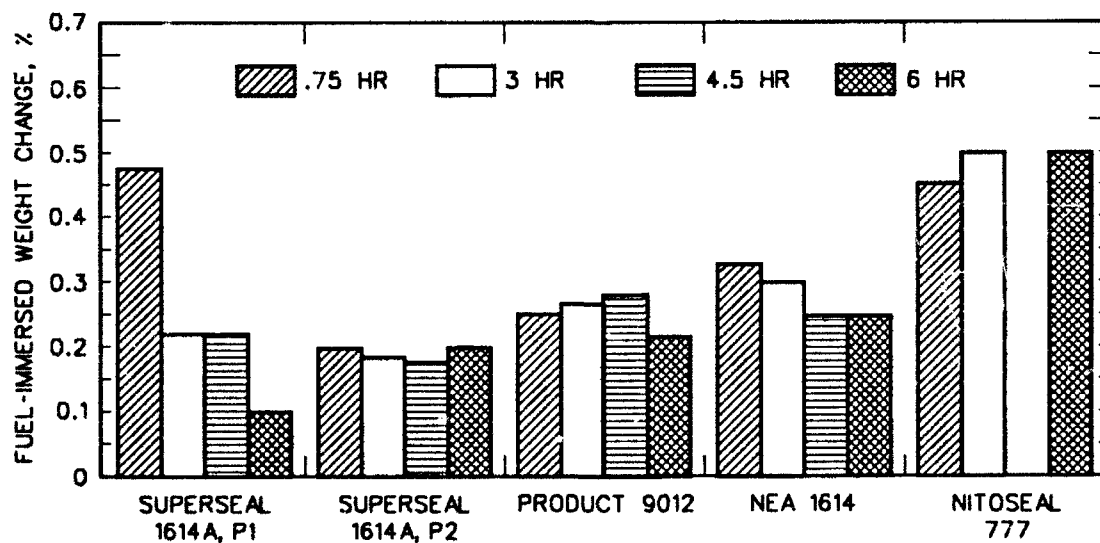


Figure 4. Fuel-immersed weight change of jet-fuel-resistant sealants after 0.75, 3, 4.5, and 6 hr of heating

unsatisfactory, but overall, Product 9012 appeared to be less affected by extended heating times than the Superseal 1614A samples.

29. The NEA 1614 sealant met the specification test requirements of all four heating times. The nonimmersed penetration values indicated a softening of the material as the heating time was extended from 45 min to 4.5 hr, but then hardened after heating time of 6.0 hr. The fuel-immersed penetration values indicated a slight hardening as the heating time was extended. The fuel-immersed change-in-weight values decreased as the heating time increased, but the flow and bond to concrete results did not vary.

30. The Nitoseal 777 did not conform to FS SS-S-1614A test requirements at heating times of 45 min or 3.0 hr as reported by Crafco, but it did conform to the testing requirements at the 6.0 hr heating time. The 4.5 hr heating time testing was not conducted due to insufficient material. The nonimmersed penetration values exhibited a decrease as the heating time increased, indicating that the sealant hardened as it was heated. The fuel-immersed penetration values exhibited an increase from the 45 min heating time to 3.0 hr and a decrease after 6.0 hr. The fuel-immersed change-in-weight values did not change appreciably during heating, and the flow and bond to concrete results did not change.

31. The specification testing at the various heating times indicate that different sealants are affected to different degrees by extended heating. The results, however, do not infer that one material will perform better in the field than the other sealants.

Additional Laboratory Testing

32. In order to further evaluate the physical characteristics of the fuel-resistant sealants, additional testing was performed. The additional testing procedures were selected to provide a clearer indication of the performance level or physical limits of the available JFR sealants by testing beyond FS SS-S-1614A procedures. Additional tests currently used for sealants meeting other specifications were used to identify levels of high temperature performance, low temperature performance, and elastic characteristics. The additional tests were performed on sealants which were heated for various time periods to determine the effects of heating on each material. Since most of

the JFR sealants were supplied as liquids which polymerize during heating, testing of the liquid materials in their as supplied state was also performed.

33. Test procedures used to determine "as-supplied" properties were as follows:

- a. Brookfield viscosity at 77°F and 100°F. These evaluations were performed to determine the thickness of each material at typical ambient temperatures which the sealant may experience prior to application. The viscosity indicates the ease with which the material may be handled when adding to melter-applicator devices. Testing was performed using a Brookfield Model HAT viscometer, with a No. 4 probe at 20 rpm for $77 \pm 1^\circ\text{F}$ testing, and a No. 2 probe at 20 rpm for $100 \pm 1^\circ\text{F}$ testing.
- b. Oven-aged weight change at 158°F. The weight change was evaluated in two different sample configurations to determine the volatile content of the sealant materials. The first sample configuration was a standard 6 oz (70 mm diam, and 45 mm deep) ointment tin that is used for penetration testing in FS SS-S-1614A. For this evaluation, 100 ± 1 g of the sealant was placed in the tin. The second configuration was a 140-mm diam by 9.5-mm deep pan as specified in ASTM D1754 with 100 ± 1 g of sealant. For each configuration, the pans with sealant were placed in a $158^\circ\text{F} \pm 2^\circ\text{F}$ forced-draft oven and their weight was monitored after 0, 2, 6, 24, 72, and 168 hr of exposure. From these measurements, percent weight loss was calculated.

34. Test procedures used to determine additional physical properties were as follows:

- a. Resilience. Resilience was determined after four different curing periods used in several different sealant testing procedures. The resilience parameter provides an indication of the elastic characteristics of the sealant. Evaluation at several different curing periods provides an indication of variations which may occur in elastic characteristics during aging. Testing was performed in accordance with FS SS-S-1401C except four different curing and conditioning periods prior to conditioning in the 77°F water bath were used. The four curing and conditioning periods were (a) 24 ± 2 hr at standard conditions (the standard FS SS-S-1401C procedure), (b) 72 ± 4 hr at standard conditions (the standard ASTM D3583 procedure), (c) 72 ± 4 hr at standard conditions followed by 24 ± 2 hr in a $158^\circ \pm 2^\circ\text{F}$ forced-draft oven (the standard ASTM D3583 aged procedure), and (d) 72 ± 4 hr at standard conditions followed by 168 ± 4 hr in a $158^\circ \pm 2^\circ\text{F}$ forced-draft oven (the FS SS-S-1401C aged procedure).
- b. Cone penetration at 0°F. This test provides an indication of the material stiffness at 0°F. The test procedure consisted of further conditioning the 77°F penetration samples from standard FS SS-S-1614A testing in an air atmosphere at $0 \pm 2^\circ\text{F}$ for 16 ± 4 hr immediately after determining the 77°F penetration. The specimen was removed from the 0°F atmosphere and tested

immediately at standard lab conditions for penetration using the 150 g cone with a 5 sec duration. Three determinations were made within 60 sec from the time of removal from the 0°F atmosphere and the results were averaged.

- c. Flow testing. Flow of the sealants was evaluated at higher temperatures and longer times using ASTM D3583 and FS SS-S-200E procedures. This testing was conducted to provide an indication of high service temperature flow resistance properties. Testing was performed using samples prepared in accordance with FS SS-S-1614A procedures. Samples were then exposed to the following four sets of conditions to determine flow:
(a) $158^{\circ} \pm 2^{\circ}\text{F}$ for 5 hr, (b) $158^{\circ} \pm 2^{\circ}\text{F}$ for 24 hr,
(c) $200^{\circ} \pm 2^{\circ}\text{F}$ for 5 hr, and (d) $200^{\circ} \pm 2^{\circ}\text{F}$ for 24 hr.
- d. Oven-aged weight loss, 158°F . This evaluation was performed after 24, 72, and 168 hr of exposure to determine sealant volatile loss after application due to exposure to summer temperatures. The test consisted of pouring specimens into tared 6 oz (70 mm diam \times 45 mm deep) tins, curing at standard laboratory conditions for 72 ± 4 hr, weighing each to the nearest one hundredth of a gram, and then placing them in a $158^{\circ} \pm 2^{\circ}\text{F}$ forced-draft oven. Specimens were then removed after 24, 72, and 168 hr, allowed to cool for 1 hr, and weighed. Percent weight loss for each condition was calculated.
- e. Bond testing. Bond testing was performed using three additional procedures which consisted of higher extensions and lower temperatures than standard FS SS-S-1614A procedures. This testing was conducted to provide a better indication of the low temperature stiffness and adhesion properties of each sealant. Only nonimmersed bond specimens were tested using the additional procedures. Specimen preparation was the same as specified in FS SS-S-1614A. Testing conditions for three cycles each were as follows: (a) $0 \pm 2^{\circ}\text{F}$, 100 percent extension, (b) $-20 \pm 2^{\circ}\text{F}$, 50 percent extension, and (c) $-20 \pm 2^{\circ}\text{F}$, 100 percent extension. The same evaluation criteria for 1614A nonimmersed bond testing was used for evaluating the bond tests.
- f. Flame resistance. Testing was performed using the FS SS-S-200E procedures to provide an indication of the resistance of each sealant to aircraft exhaust.
- g. Tack free time. Testing was performed to determine cure time and to provide an estimate of the tack free time using the basic FS SS-S-200E procedure.
- h. Brookfield viscosity. The viscosity was performed with the heated material at the safe heating temperature ($\pm 2^{\circ}\text{F}$) just prior to pouring specimens. The viscosity testing was conducted to provide an indication of sealant application characteristics. Testing was performed using a Brookfield Model HAT viscometer with the appropriate probes and speeds (typically No. 4 at 20 rpm).

- i. Stress relaxation. Stress relaxation testing was performed on the sealants conditioned at each heating period. The testing was conducted at both $76 \pm 1^\circ\text{F}$ and $-20 \pm 2^\circ\text{F}$. The testing consisted of extending bond specimens prepared in accordance with FS SS-S-1614A to a predetermined distance or force using an extension frame. The force decay curve as a function of time was then monitored. Before testing, the bond specimens were conditioned for a minimum of 4 hr at the test temperature. For testing at 76°F , specimens were placed in the extension device grips, and then extended at a uniform rate in 10 sec to 100 percent, (1.00 in. separation between the blocks). The initial force was then recorded and time measurements were started as soon as the 100 percent extension was reached. Extension was then maintained at 100 percent, and the force recorded as the specimen relaxes at 10, 30, and 60 sec, as well as 5, 10, and 60 min. For testing at -20°F , the procedure was similar, except the specimens were loaded to a force of 200 lb, (50 psi) in the 10 sec period instead of 100 percent. The force relaxation was then monitored at 0, 30, and 60 sec, 2, 5, 30, and 60 min, and 3 and 6 hr. During the test, the force was monitored using a calibrated 100 lb force ring for 76°F tests, and a 500-lb force ring for -20°F tests. A diagram of the extension device used is shown in Figure 5. Force data were converted into pounds per square inch based on the original specimen surface area of 4.00 sq in.

35. Additional test result data are summarized in Tables 6 through 11 and plotted in Figures 6 through 21.

Additional Laboratory Test Analysis

36. The "as-supplied" properties of the liquid JFR sealants are provided in Table 6. The Product 9012 as-supplied properties were not determined since it was supplied in solid form.

Brookfield viscosity

37. The Brookfield viscosities of the liquid sealants decreased as the materials were heated to 100°F as illustrated in Figure 6. As temperature increases above 100°F to approximately $180\text{-}200^\circ\text{F}$, the viscosity decreases. Then the liquid materials get to an extremely high viscosity at 200 to 230°F , and then they thin down to the viscosity at the safe heating temperature. The viscosity of each sealant significantly increased as the temperature was increased from 100°F to the safe heating temperature. The viscosity continued to increase as the sealant was exposed to extended heating times. The increase in viscosity at the extended heating times indicates that the sealants become more difficult to pump out of the application equipment as

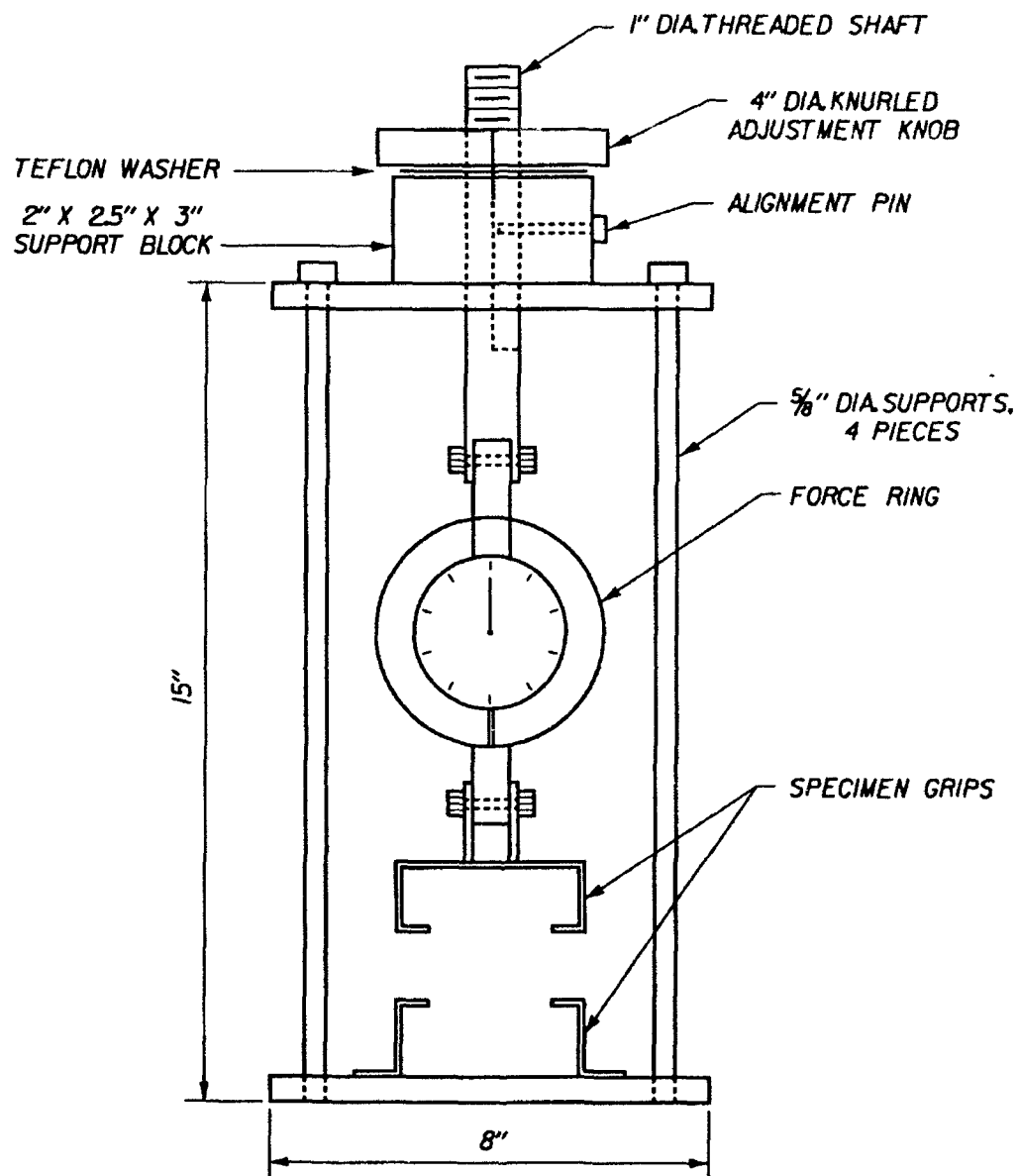


Figure 5. Force-extension device used for stress relaxation determinations

Table 6
"As Supplied" Properties of Jet-Fuel-Resistant Sealants

Parameter	Superseal 1614A, P1*	Superseal 1614A, P2**	Product 9012	NEA 1614	Nitoseal 777
Brookfield viscosity, cps					
77°F (No. 4 at 20 rpm)	5,800	9,400	†	14,800	7,400
100°F (No. 2 at 20 rpm)	1,600	1,640	--	2,000	1,920
Oven aged weight change, 6 oz container, 158°F, percent					
2 hr	-0.3	-0.4	--	-0.3	-0.1
6 hr	-0.5	-0.8	--	-0.4	-0.3
24 hr	-1.0	-1.1	--	-0.8	-0.6
72 hr	-1.6	-1.8	--	-1.6	-1.6
168 hr	-2.4	-2.2	--	-1.9	-2.9
Oven aged weight change, 5 1/2 in. pan, 158°F, percent					
2 hr	-0.9	-0.9	--	-0.5	-0.4
6 hr	-1.4	-1.3	--	-0.8	-1.0
24 hr	-3.0	-2.8	--	-1.9	-3.0
72 hr	-4.8	-4.2	--	-3.4	-6.4
168 hr	-7.0	-5.4	--	-5.0	-9.8

* Material from Plant 1.

** Material from Plant 2.

† Material is solid, therefore properties were not determined.

Table 7
Additional Physical Characteristics
Crafco Superseal 1614A, Plant 1

Test Parameter	Heating Period			
	45 min	3.0 hr	4.5 hr	6.0 hr
Resilience				
24 hr, percent	41	42	41	48
72 hr, percent	53	57	54	60
Aged 72 hr resilience				
24 hr, 158°F, percent	36	27	33	34
168 hr, 158°F, percent	21	27	17	22
Penetration, 0°F, cm				
Nonimmersed	0.16	0.18	0.13	0.07
Fuel-immersed	0.22	0.26	0.17	0.16
Flow, cm				
158°F, 5 hr	0.0	0.0	0.0	0.0
158°F, 24 hr	0.0	0.0	0.0	0.0
200°F, 5 hr	5.0+	5.0+	5.0+	0.9
200°F, 24 hr	5.0+	5.0+	5.0+	1.9
Oven aged weight loss, percent				
24 hr, 158°F	-0.5	-0.4	-0.4	-0.3
72 hr, 158°F	-0.7	-0.7	-0.6	-0.6
168 hr, 158°F	-1.3	-1.1	-1.0	-0.9
Nonimmersed bond				
0°F, 100 percent, 3 cycles	S*	S	S	U**
-20°F, 50 percent, 3 cycles	U	U	U	U
-20°F, 100 percent, 3 cycles	U	U	U	U
Flame resistance	U	U	U	U
Tack free time, min	35	15	8	6
Viscosity, Brookfield, 270°F, cps	7,000	7,200	8,400	10,400

* S signifies satisfactory performance in accordance with the test specification.

** U signifies unsatisfactory performance.

Table 8
Additional Physical Characteristics
Crafco Superseal 1614A, Plant 2

Test Parameter	Heating Period			
	45 min	3.0 hr	4.5 hr	6.0 hr
Resilience				
24 hr, percent	58	54	54	57
72 hr, percent	65	63	64	67
Aged 72 hr resilience				
24 hr, 158°F, percent	57	39	31	44
168 hr, 158°F, percent	30	29	32	30
Penetration, 0°F, cm				
Nonimmersed	0.17	0.12	0.11	0.07
Fuel-immersed	0.28	0.19	0.12	0.09
Flow, cm				
158°F, 5 hr	0.0	0.0	0.0	0.0
158°F, 24 hr	0.0	0.0	0.0	0.0
200°F, 5 hr	0.0	0.0	0.3	0.3
200°F, 24 hr	0.0	0.2	0.4	0.3
Oven aged weight loss, percent				
24 hr, 158°F	-0.5	-0.3	-0.2	-0.2
72 hr, 158°F	-0.9	-0.5	-0.7	-0.4
168 hr, 158°F	-1.0	-0.9	-0.7	-0.6
Nonimmersed bond				
0°F, 100 percent, 3 cycles	S*	U**	U	U
-20°F, 50 percent, 3 cycles	U	U	U	U
-20°F, 100 percent, 3 cycles	U	U	U	U
Flame resistance	U	U	U	U
Tack free time, min	9	8	6	5
Viscosity, Brookfield, 270°F, cps	7,200	7,920	11,040	12,300

* S signifies satisfactory performance in accordance with the test specification.

** U signifies unsatisfactory performance.

Table 9
Additional Physical Characteristics
Product 9012

Test Parameter	Heating Period			
	45 min	3.0 hr	4.5 hr	6.0 hr
Resilience				
24 hr, percent	38	49	52	54
72 hr, percent	53	58	59	57
Aged 72 hr resilience				
24 hr, 158°F, percent	45	49	58	51
168 hr, 158°F, percent	27	29	30	28
Penetration, 0°F, cm				
Nonimmersed	0.10	0.09	0.08	0.08
Fuel-immersed	0.19	0.15	0.16	0.16
Flow, cm				
158°F, 5 hr	0.0	0.0	0.0	0.0
158°F, 24 hr	0.0	0.0	0.0	0.0
200°F, 5 hr	0.5	0.5	0.6	0.6
200°F, 24 hr	0.6	0.5	0.6	0.6
Oven aged weight loss, percent				
24 hr, 158°F	-0.3	-0.5	-0.2	-0.2
72 hr, 158°F	-0.5	-0.4	-0.43	-0.5
168 hr, 158°F	-0.8	-0.8	-0.6	-0.6
Nonimmersed bond				
0°F, 100 percent, 3 cycles	U*	U	U	U
-20°F, 50 percent, 3 cycles	U	U	U	U
-20°F, 100 percent, 3 cycles	U	U	U	U
Flame resistance	U	U	U	U
Tack free time, min	20	15	9	7
Viscosity, Brookfield, 280°F, cps	8,400	11,000	12,400	14,400

* U signifies unsatisfactory performance in accordance with the test specification.

Table 10
Additional Physical Characteristics
NEA 1614

Test Parameter	Heating Period			
	45 min	3.0 hr	4.5 hr	6.0 hr
Resilience				
24 hr, percent	51	50	56	51
72 hr, percent	50	61	63	60
Aged 72 hr resilience				
24 hr, 158°F, percent	41	50	39	47
168 hr, 158°F, percent	29	35	30	27
Penetration, 0°F, cm				
Nonimmersed	0.12	0.13	0.08	0.07
Fuel-immersed	0.23	0.23	0.22	0.19
Flow, cm				
158°F, 5 hr	0.0	0.0	0.0	0.0
158°F, 24 hr	0.0	0.0	0.0	0.0
200°F, 5 hr	0.2	0.0	0.0	0.0
200°F, 24 hr	0.3	0.1	0.1	0.1
Oven aged weight loss, percent				
24 hr, 158°F	-0.3	-0.3	-0.4	-0.3
72 hr, 158°F	-0.6	-0.6	-0.5	-0.5
168 hr, 158°F	-0.9	-0.9	-0.8	-0.8
Nonimmersed bond				
0°F, 100 percent, 3 cycles	U*	U	U	U
-20°F, 50 percent, 3 cycles	U	U	U	U
-20°F, 100 percent, 3 cycles	U	U	U	U
Flame resistance	U	U	U	U
Tack free time, min	20	20	12	7
Viscosity, Brookfield, 270°F, cps	9,000	12,400	13,000	16,400

* U signifies unsatisfactory performance in accordance with the test specification.

Table 11
Additional Physical Characteristics
Nitoseal 777

Test Parameter	Heating Period			
	45 min	3.0 hr	4.5 hr	6.0 hr
Resilience				
24 hr, percent	63	58	--*	55
72 hr, percent	72	71	--	64
Aged 72 hr resilience				
24 hr, 158°F, percent	44	55	--	54
168 hr, 158°F, percent	43	53	--	31
Penetration, 0°F, cm				
Nonimmersed	0.18	0.16	--	0.14
Fuel-immersed	0.14	0.15	--	0.10
Flow, cm				
158°F, 5 hr	0.0	0.0	--	0.0
158°F, 24 hr	0.0	0.0	--	0.0
200°F, 5 hr	0.1	0.1	--	0.1
200°F, 24 hr	0.1	0.1	--	0.1
Oven aged weight loss, percent				
24 hr, 158°F	-0.3	-0.3	--	-0.1
72 hr, 158°F	-0.4	-0.6	--	-0.8
168 hr, 158°F	-1.0	-1.1	--	-1.1
Nonimmersed bond				
0°F, 100 percent, 3 cycles	S**	S	--	U†
-20°F, 50 percent, 3 cycles	U	U	--	U
-20°F, 100 percent, 3 cycles	U	U	--	U
Flame resistance	U	U	--	U
Tack free time, min	15	15	--	10
Viscosity, Brookfield, 300°F, cps	2,600	3,200	--	4,700

* Testing was not performed after 4.5 hr of heating.

** S signifies satisfactory performance in accordance with the test specification.

† U signifies unsatisfactory performance.

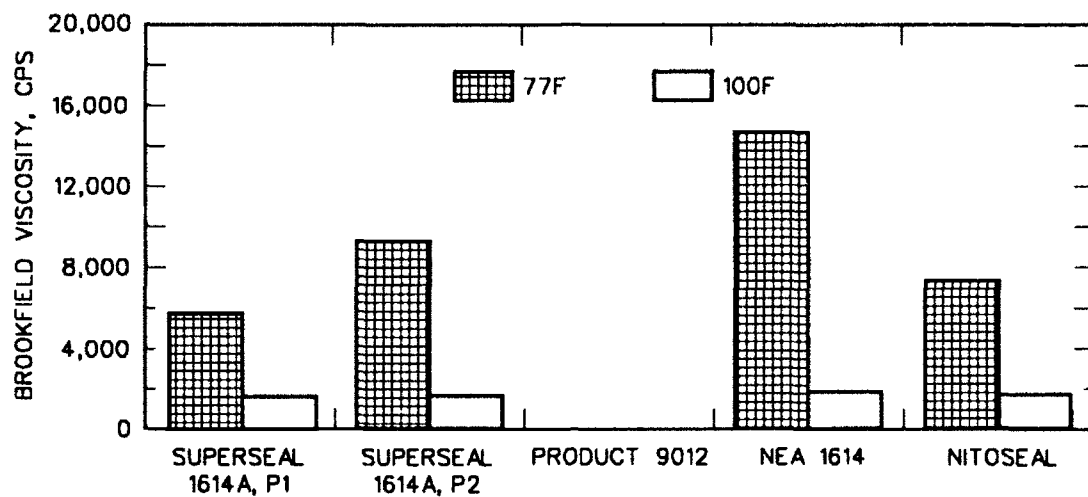


Figure 6. Viscosity at 77°F and 100°F for liquid jet-fuel-resistant sealants

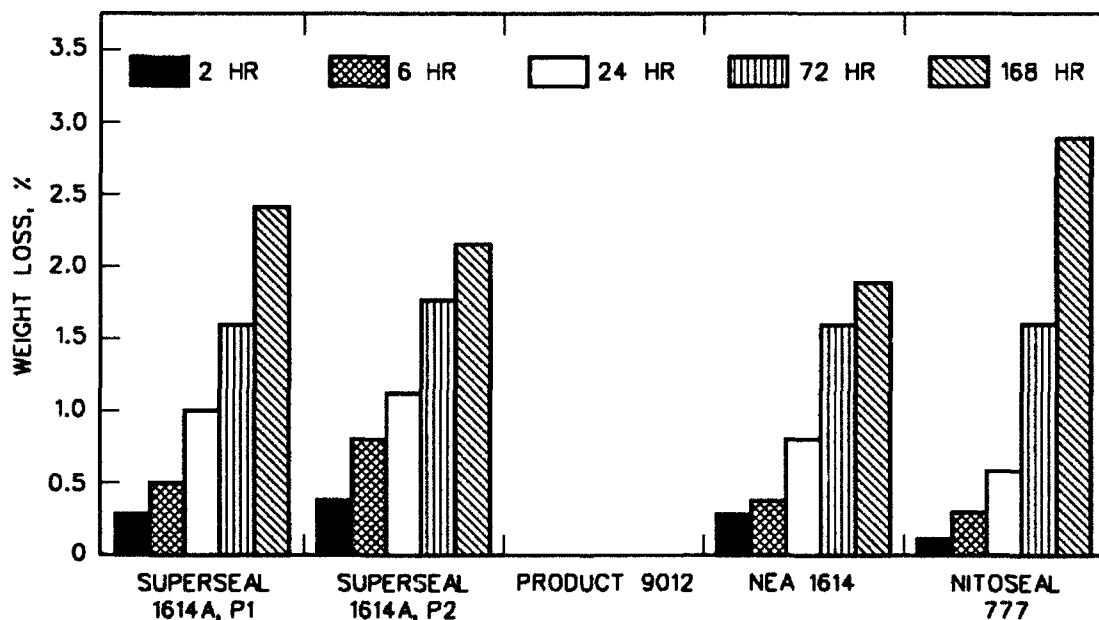


Figure 7. Weight loss at 158°F using a 6-oz. container for liquid jet-fuel-resistant sealants

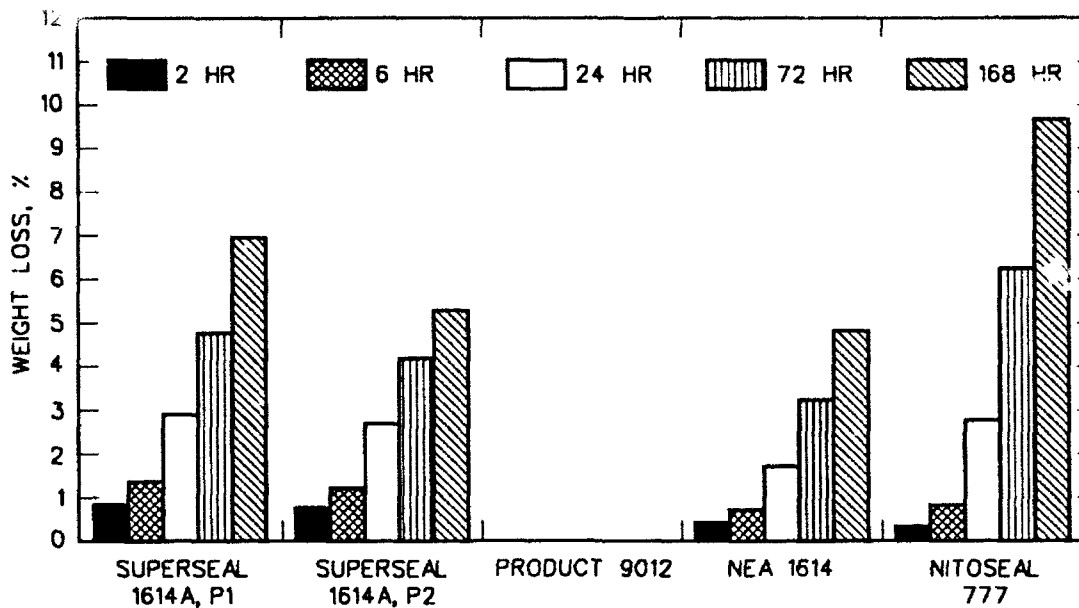


Figure 8. Weight loss at 158°F using a 140mm diameter pan for liquid jet-fuel-resistant sealants

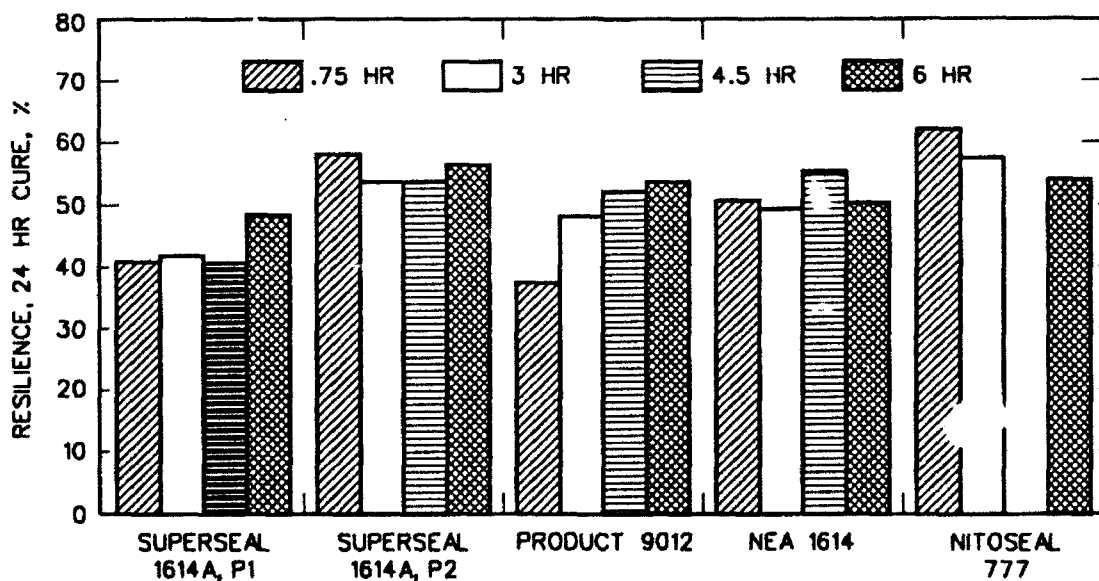


Figure 9. Twenty-four hr cured resilience of jet-fuel-resistant sealants after 0.75, 3, 4.5, and 6 hr of heating

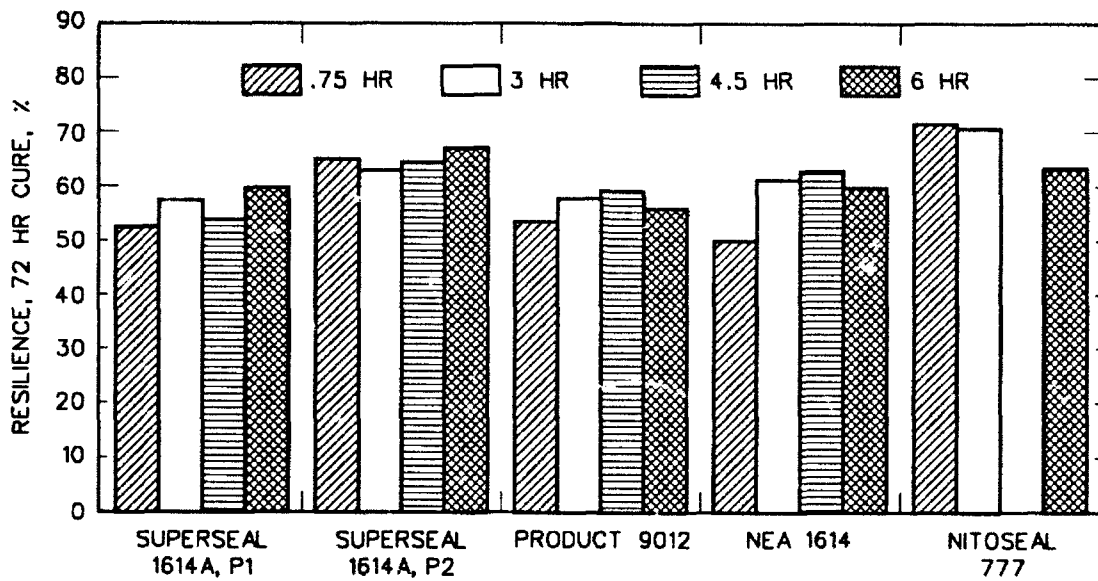


Figure 10. Seventy-two hr cured resilience of jet-fuel-resistant sealants after 0.75, 3, 4.5, and 6 hr of heating

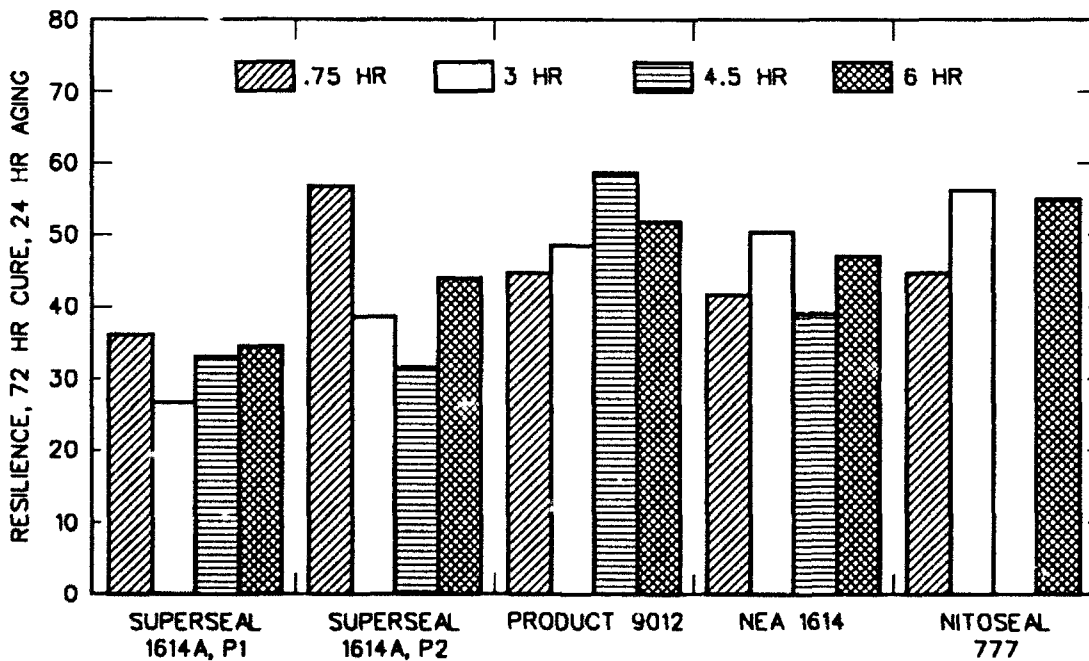


Figure 11. Seventy-two hr cured, 24 hr aged resilience of jet-fuel-resistant sealants after 0.75, 3, 4.5, and 6 hr of heating

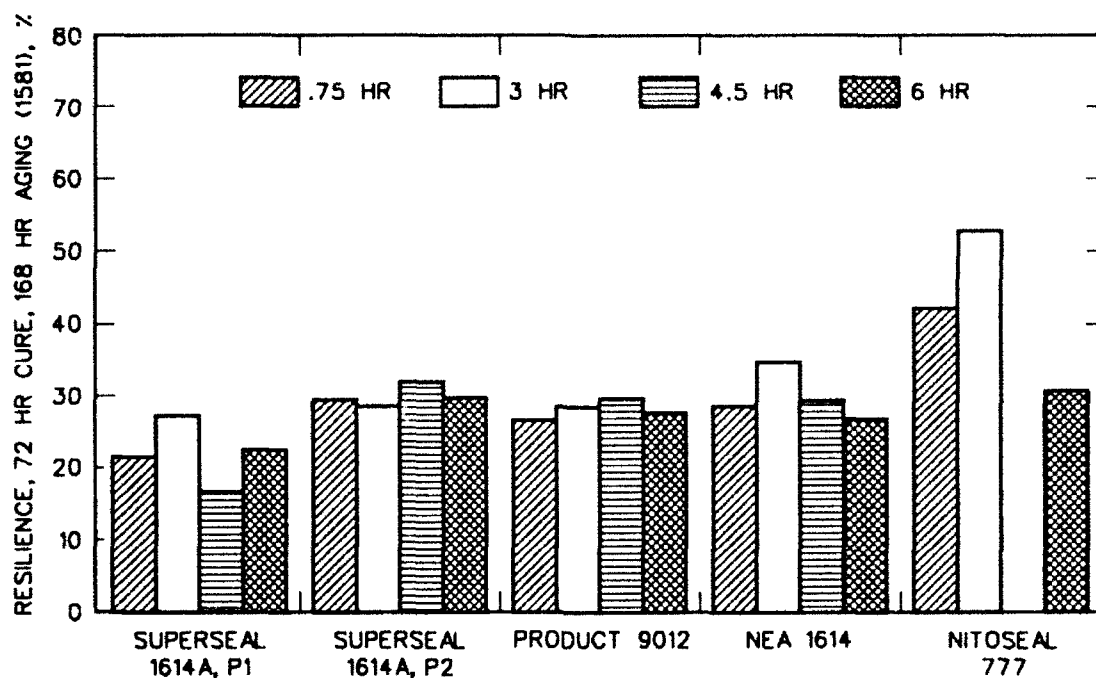


Figure 12. Seventy-two hr cured, 168 hr aged resilience of jet-fuel-resistant sealants after 0.75, 3, 4.5, and 6 hr of heating

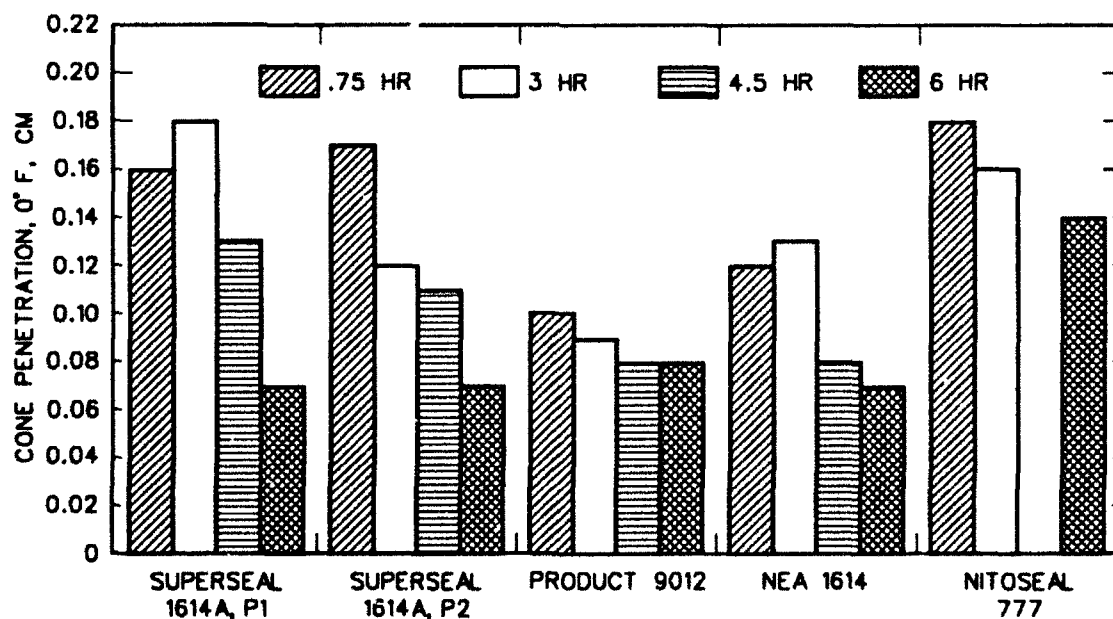


Figure 13. Cone penetration at 0°F for jet-fuel-resistant sealants after 0.75, 3, 4.5, and 6 hr of heating

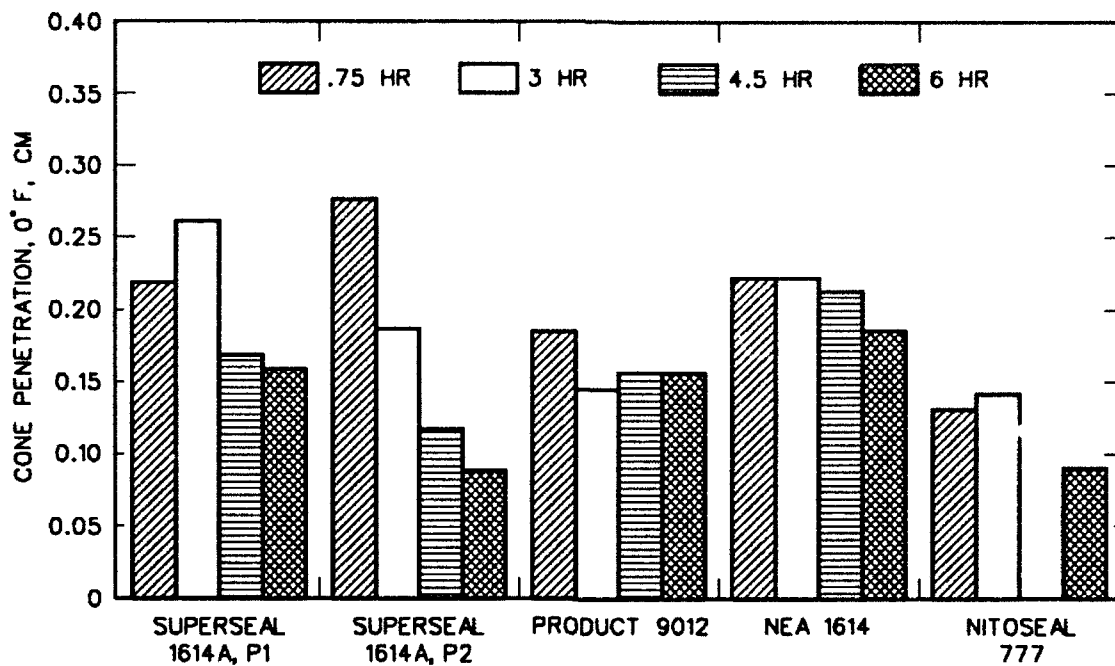


Figure 14. Fuel-immersed cone penetration at 0°F for jet-fuel-resistant sealants after 0.75, 3, 4.5, and 6 hr of heating

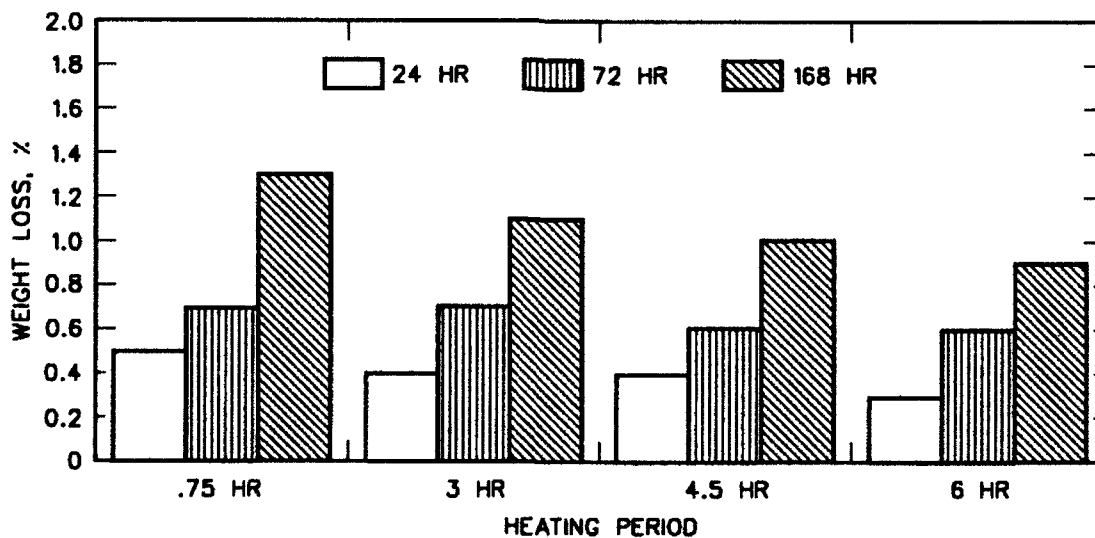


Figure 15. Weight loss for superseal 1614A, Plant 1 after 24, 72, and 168 hr of aging at 158°F

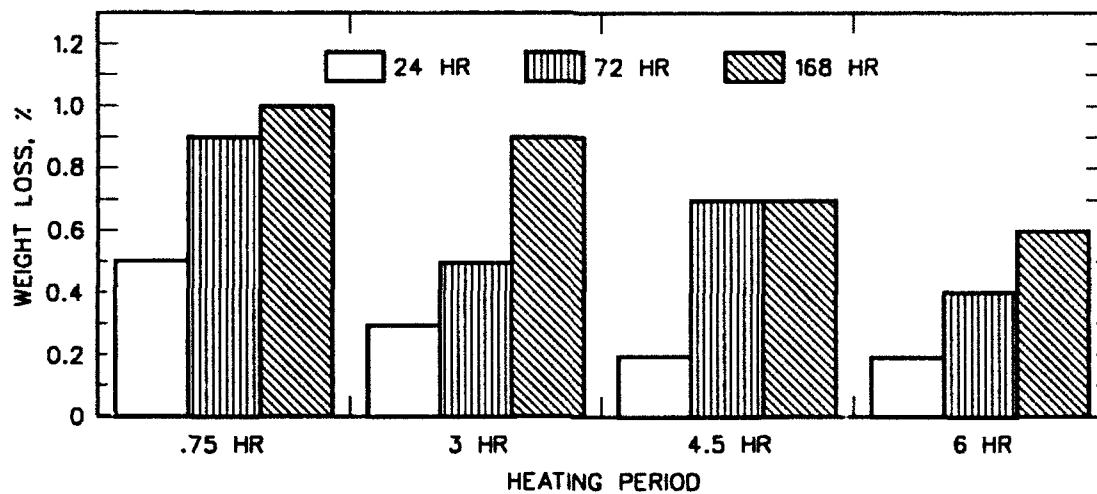


Figure 16. Weight loss for superseal 1614A, Plant 2 after 24, 72, and 168 hr of aging at 158°F

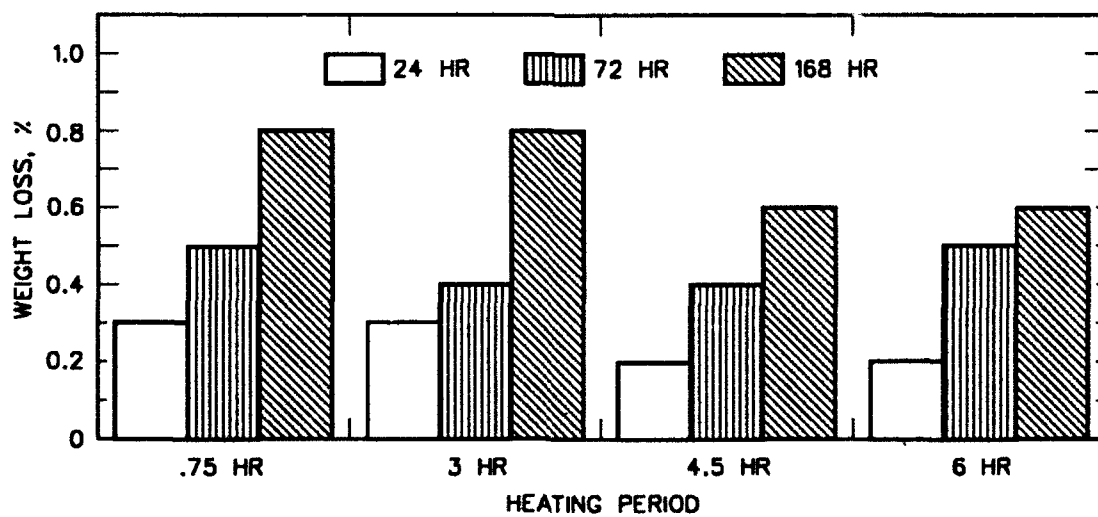


Figure 17. Weight loss for product 9012 after 24, 72, and 168 hr of aging at 158°F

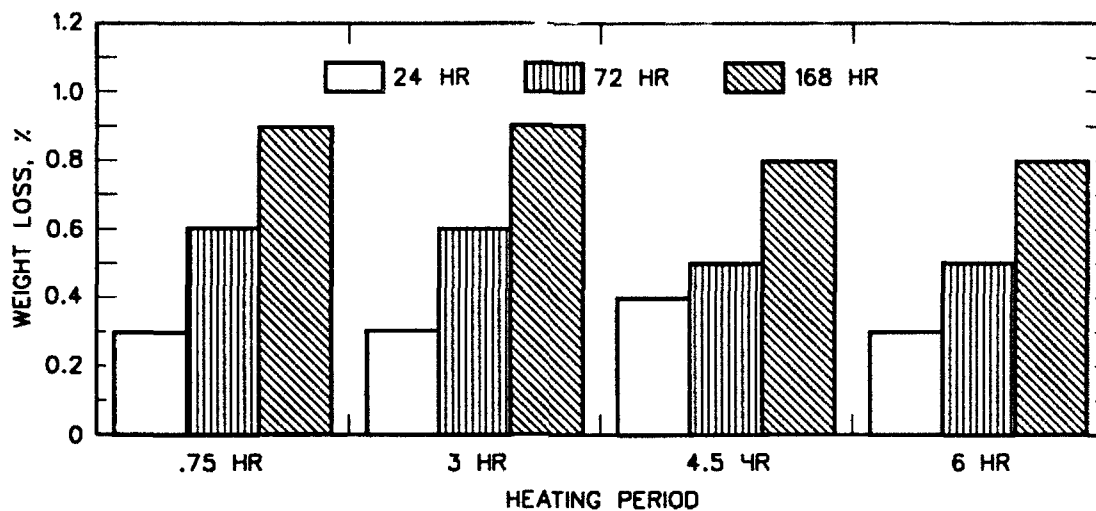


Figure 18. Weight loss for NEA 1614 after 24, 72, and 168 hr of aging at 158°F

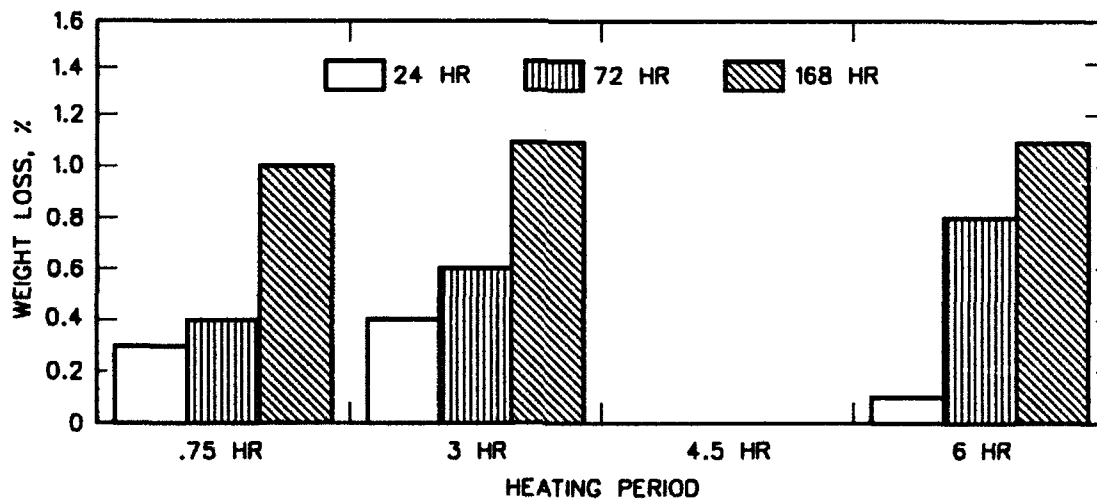


Figure 19. Weight loss for Nitoseal 777 after 24, 72, and 168 hr of aging at 158°F

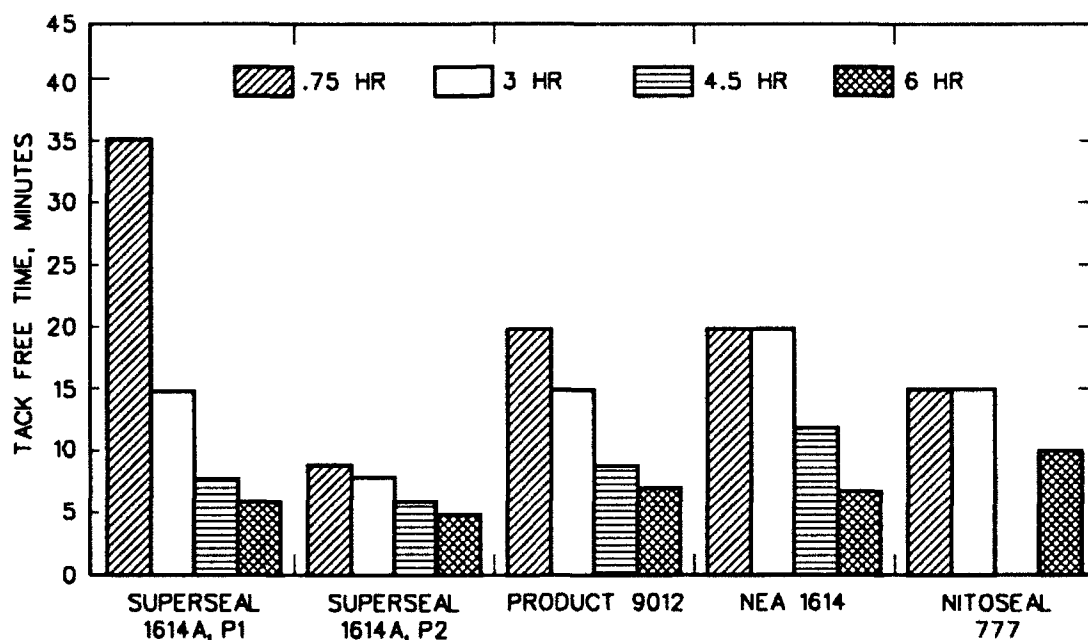


Figure 20. Tack free time of jet-fuel-resistant sealants after 0.75, 3, 4.5, and 6 hr of heating

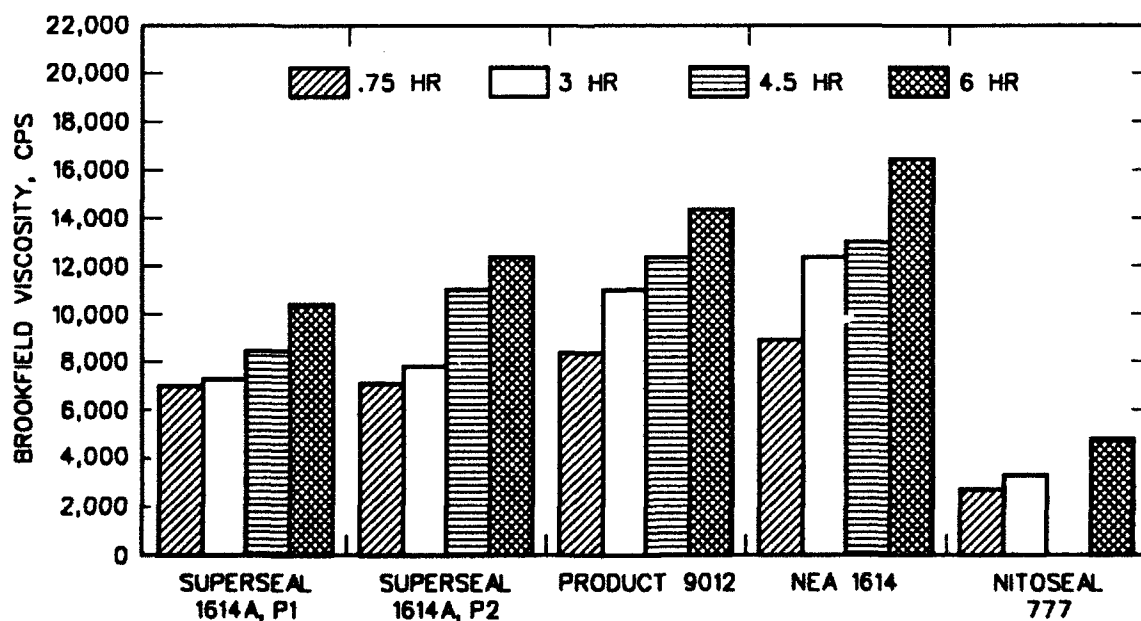


Figure 21. Viscosity at safe heating temperature for jet-fuel-resistant sealants after 0.75, 3, 4, 4.5, and 6 hr of heating

heating times are increased. Tables 7-11 provide the additional test results for the sealants.

Oven-aged change in weight

38. The oven-aged change-in-weight data for the as-received sealants indicate they all experienced weight loss with aging. The tests conducted in the 140-mm-diam pan (Figure 8) exhibited greater weight loss than the tests conducted in the 6-oz tin (Figure 7). The greater weight loss occurs in the 140-mm-diam samples due to the greater surface area exposed to the heated air. The oven-aged change-in-weight tests indicate that the Nitoseal 777 had the largest volatile loss and the NEA 1614 experienced the smallest loss. The oven-aged change-in-weight results for the sealants exposed to the various heating times are plotted in Figures 15 through 19. The test results indicated that as the sealants were exposed to longer heating times before conditioning, the percent weight loss decreased except for the Nitoseal 777. This trend was expected, since the sealants were exposed to longer heating times and the volatiles would be lost during the heating.

39. The maximum weight loss after 168 hr of aging was 1.3 percent for Superseal 1614A, Plant 1 after 45 min of heating, and the minimum weight loss was 0.6 percent for Superseal 1614A, Plant 2 after 6.0 hr of heating. The two sealants manufactured using raw materials from different sources again exhibited different physical properties.

40. During the oven-age testing, the surface of the sealant samples became brittle. An increase in brittleness or hardness also occurred as the samples were exposed to longer aging periods. This phenomena was studied in greater detail, and the testing procedures and results are discussed in Part V.

Resilience testing

41. Resilience after 24 hr of curing at ambient conditions (Figure 9) varied from a low of 38 percent for the Product 9012 after 45 min of heating to a high of 63 percent for Nitoseal 777 after 45 min of heating. Resilience data varied depending on heating period, and differences exist between sealants. Superseal 1614A, Plant 1 had the lowest overall average resilience (43 percent), and Superseal 1614A, Plant 2 had the highest average (57 percent). Superseal 1614A, Plant 2 experienced the least change during heating (4 percent range of data), and Product 9012 experienced the greatest change (16 percent range of data).

42. Resilience after 72 hr of curing at ambient conditions (Figure 10) varied from a low of 50 percent for NEA 1614 after 45 min of heating to a high of 72 percent for Nitoseal 777 after 45 min of heating. Superseal 1614A, Plant 1 had the lowest average resilience (56 percent) and Nitoseal 777 had the highest average (69 percent). All resilience results for the sealant materials were higher after 72 hr of curing than after 24 hr of curing. The one exception to this trend was the NEA 1614 that had been exposed to 45 min of heating. The average increase in resilience varied from 6 to 13 percent.

43. The resilience of the sealants after they were subjected to aging at 158°F for 24 hr (Figure 11) varied from a low of 27 percent for Superseal 1614A, Plant 1 after a 3 hr heating time to a high of 58 percent for Product 9012 after a 4.5 hr heating time. Superseal 1614A, Plant 1 had the lowest overall average (33 percent), and Product 9012 and Nitoseal 777 had the highest averages (51 percent). All resilience results for the sealant materials were lower after aging for 24 hr at 158°F than the sealants cured for 72 hr. The greatest overall average decrease in resilience was for Superseal 1614A, Plant 1 (24 percent decrease), and the least decrease was for Product 9012 (6 percent).

44. The resilience of the sealants after they were subjected to aging at 158°F for 168 hr (Figure 12) varied from a low of 17 percent for Superseal 1614A, Plant 1 after 4.5 hr of heating to a high of 53 percent for Nitoseal 777 after 3 hr of heating. Superseal 1614A, Plant 1 had the lowest average resilience (22 percent) and Nitoseal 777 had the highest (42 percent). In all but two tests, resilience results after 168 hr of aging were lower than at 24 hr of aging. Product 9012 experienced the greatest average decrease (22 percent) and Nitoseal 777 experienced the least (9 percent).

45. The above results and discussions indicate that resilience varies significantly between the sealants tested due to the length of heating period used and the curing and aging period. Lowest overall resilience results were obtained for Superseal 1614A, Plant 1 (39 percent), and the highest results were for Nitoseal 777 (59 percent). The effect of heating period varies depending on the sealant tested. For some materials heating had little effect on results, while with others, the results tended to either increase or decrease with heating. Increasing curing time from 24 to 72 hr prior to testing caused the resilience to increase by an average of 9 percent for all materials tested. The resilience values within a specific heating for the

sealants exhibited a general decrease as the samples were exposed to aging, but clear trends were not exhibited for specific resilient testing of varying heating times.

Penetration testing

46. Penetration testing at 0°F (Figure 13) showed a decrease as heating time increased for all sealants. Results after 45 min of heating varied from 0.10 to 0.18 cm. Results after 6 hr of heating varied from 0.07 to 0.14 cm. All materials tested had lower penetrations after 6 hr than after 45 min. Percent retained penetration after 6 hr compared to 45 min of heating varied from a low of 41 percent for Superseal 1614A, Plant 2 to 80 percent for Product 9012.

47. Fuel-immersed penetration data at 0°F are plotted in Figure 14. As with nonimmersed penetration at 0°F, results show a decrease as the heating time was increased. Additionally for all materials except Nitoseal 777, fuel-immersed penetration results at 0°F are higher than nonimmersed results. This may be due to a softening effect of fuel with some materials. After 45 min of heating, Superseal 1614A, Plant 2 had the highest penetration (0.28 cm), and Nitoseal 777 had the lowest (0.14 cm). After 6 hr of heating, NEA 1614 had the highest penetration (0.19 cm) and Superseal 1614A, Plant 2 had the lowest (0.09 cm). Retesting for 0°F fuel-immersed penetration of specimens which were stored several days at standard conditions showed decreases in penetration. It is believed this was caused by the evaporation of fuel from the samples. The penetration values indicate that the sealants became more brittle with aging.

Flow testing

48. The flow for all of the sealants at 158°F for 5 and 24 hr was 0.0 cm at all heating periods. Testing at 200°F resulted in a wide variation in flow results. Superseal 1614A, Plant 1 flowed more than 5.0 cm for each heat period except 6 hr, where the flow was 0.9 and 1.0 cm. The flow of all other materials was less than 1.0 cm for all heat periods. Based on this data, it appears that the current FS SS-S-1614A specification flow testing procedure could be changed to use higher temperatures for a more stringent evaluation of sealants.

Nonimmersed bond testing

49. All of the sealants from each of the heating periods failed the -20°F nonimmersed bond testing at both 50 and 100 percent extension. All

materials failed during the first extension cycle. Results obtained at 0°F using 100 percent extension varied for the different sealants and heating periods. Superseal 1614A, Plant 1 showed satisfactory results for each heating period except 6.0 hr. Superseal 1614A, Plant 2 results were satisfactory after 45 min of heating, but unsatisfactory for the other heating periods. Product 9012 and NEA 1614 results were unsatisfactory at all heating periods. Nitoseal 777 showed satisfactory results after 45 min and 3.0 hr of heating, but unsatisfactory results after 6.0 hr. Previously discussed bond results in accordance with FS SS-S-1614A (which used 50 percent extension at 0°F) showed satisfactory results for all materials. These results indicate that the FS SS-S-1614A bond testing procedure evaluates available sealant materials at or near their limit of low temperature performance.

Flame resistance testing

50. Flame resistance testing yielded unsatisfactory results for all materials tested. During the test, all materials dripped before the end of the 2 min 500°F flame exposure period which illustrates that these materials are not resistant to concentrated aircraft exhaust.

Tack free testing

51. Tack free time (Figure 20) varied from a high of 35 min for Superseal 1614A, Plant 1 to a low of 5 min of Superseal 1614A, Plant 2. Results for all materials decreased with longer heating periods. Thus, areas sealed with these materials can be opened to traffic within a short period of time without damaging the sealant.

Stress relaxation testing

52. Stress relaxation results at 76 and -20°F are summarized in Tables 12 through 15. Results are plotted in Figures 22 through 25. A plot of relaxation data at 76°F for each sealant after 3 hr of heating is shown in Figure 26. The initial stress is the highest and as time increases, stress decreases as the specimen relaxes. Since testing at 76°F was performed using a 100 percent extension, the data can also be referred to as a static modulus. Figure 22 shows the initial 76°F static modulus, and that the modulus increased with heating period. Superseal 1614A, Plant 1 after 45 min of heating had the lowest initial static modulus (3.0 psi), and Superseal 1614A, Plant 2 after 6 hr of heating had the highest (10.0 psi). Initial static modulus behaved in an inverse manner compared to 77°F penetration trends. Figure 23 shows static modulus at 76°F after a 60 min relaxation period.

Table 12
Stress Relaxation Data at $76 \pm 1^\circ\text{F}$ and $-20 \pm 2^\circ\text{F}$
Superseal 1614A, Plant 1

<u>Relaxation Period</u>	<u>Stress, psi</u>		
	<u>45 min*</u>	<u>3.0 hr</u>	<u>6.0 hr</u>
<u>Testing at $76 \pm 1^\circ\text{F}$</u>			
0 sec	3.0	3.3	5.1
10 sec	2.0	2.5	3.3
30 sec	1.8	2.3	2.8
60 sec	1.7	2.0	2.5
5 min	1.5	1.8	2.0
10 min	1.3	1.5	2.0
60 min	1.1	1.3	1.6
<u>Testing at $-20 \pm 2^\circ\text{F}$</u>			
0 sec	50.0	50.0	50.0
30 sec	48.3	49.5	49.1
60 sec	47.9	48.6	47.7
2 min	45.6	46.4	45.9
5 min	38.8	41.4	40.9
30 min	11.9	15.0	25.0
60 min	8.6	9.5	20.0
3 hr	7.7	9.1	18.6
6 hr	7.3	8.2	16.8

* Heating period used prior to pouring samples.

Table 13
Stress Relaxation Data at $76 \pm 1^\circ\text{F}$ and $-20 \pm 2^\circ\text{F}$
Superseal 1614A, Plant 2

<u>Relaxation Period</u>	<u>Stress, psi</u>		
	<u>45 min*</u>	<u>3.0 hr</u>	<u>6.0 hr</u>
<u>Testing at $76 \pm 1^\circ\text{F}$</u>			
0 sec	5.6	5.4	10.0
10 sec	3.6	3.8	7.0
30 sec	3.3	3.1	5.3
60 sec	3.0	2.9	4.5
5 min	2.6	2.4	3.3
10 min	2.4	2.3	3.0
60 min	2.0	1.9	2.5
<u>Testing at $-20 \pm 2^\circ\text{F}$</u>			
0 sec	Testing was not performed due to insufficient quantity of sealant		
30 sec			
60 sec			
2 min			
5 min			
30 min			
60 min			
3 hr			
6 hr			

* Heating period used prior to pouring samples.

Table 14
Stress Relaxation Data at $76 \pm 1^\circ\text{F}$ and $-20 \pm 2^\circ$
NEA 1614

<u>Relaxation Period</u>	<u>Stress, psi</u>		
	<u>45 min*</u>	<u>3.0 hr</u>	<u>6.0 hr</u>
<u>Testing at $76 \pm 1^\circ\text{F}$</u>			
0 sec	4.5	6.8	8.0
10 sec	3.3	5.0	6.3
30 sec	2.8	4.3	5.5
60 sec	2.6	3.8	5.0
5 min	2.3	3.3	4.0
10 min	2.2	3.0	3.8
60 min	2.0	2.5	3.0
<u>Testing at $-20 \pm 2^\circ\text{F}$</u>			
0 sec	50.0	50.0	50.0
30 sec	49.5	49.5	48.4
60 sec	48.4	48.4	46.8
2 min	46.3	46.8	45.3
5 min	40.6	42.8	41.8
30 min	16.9	18.7	19.2
60 min	11.9	14.8	17.3
3 hr	11.0	12.3	16.8
6 hr	11.0	12.3	16.3

* Heating period used prior to pouring samples.

Table 15
Stress Relaxation Data at $76 \pm 1^\circ\text{F}$ and $-20 \pm 2^\circ\text{F}$
Nitoseal 777

<u>Relaxation Period</u>	<u>Stress, psi</u>		
	<u>45 min*</u>	<u>3.0 hr</u>	<u>6.0 hr</u>
<u>Testing at $76 \pm 1^\circ\text{F}$</u>			
0 sec	5.0	5.9	6.6
10 sec	3.5	4.1	4.6
30 sec	3.1	3.6	4.0
60 sec	2.9	3.4	3.8
5 min	2.4	2.6	3.5
10 min	2.1	2.4	2.9
60 min	1.6	2.0	2.4
<u>Testing at $-20 \pm 2^\circ\text{F}$</u>			
0 sec	50.0	50.0	50.0
30 sec	49.3	48.8	49.3
60 sec	48.4	47.9	46.5
2 min	46.1	45.6	43.8
5 min	39.7	38.8	37.4
30 min	7.8	8.2	8.7
60 min	5.9	7.3	8.2
3 hr	5.9	6.8	7.6
6 hr	5.5	6.8	7.6

* Heating period used prior to pouring samples.

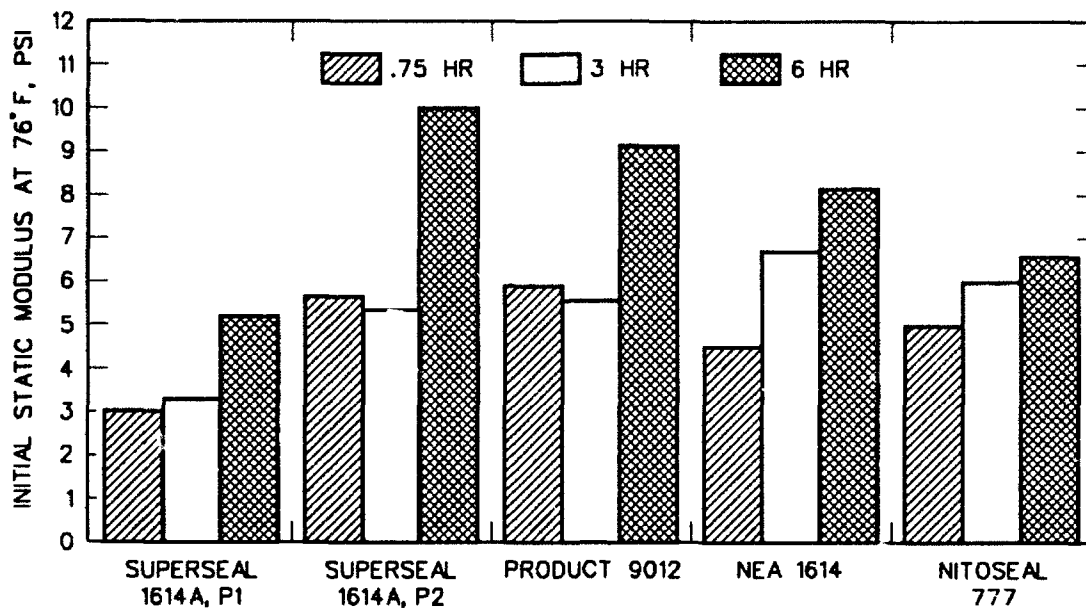


Figure 22. Initial static modulus at 100 percent extension at 76°F for jet-fuel-resistant sealants after 0.75, 3, and 6 hr of heating

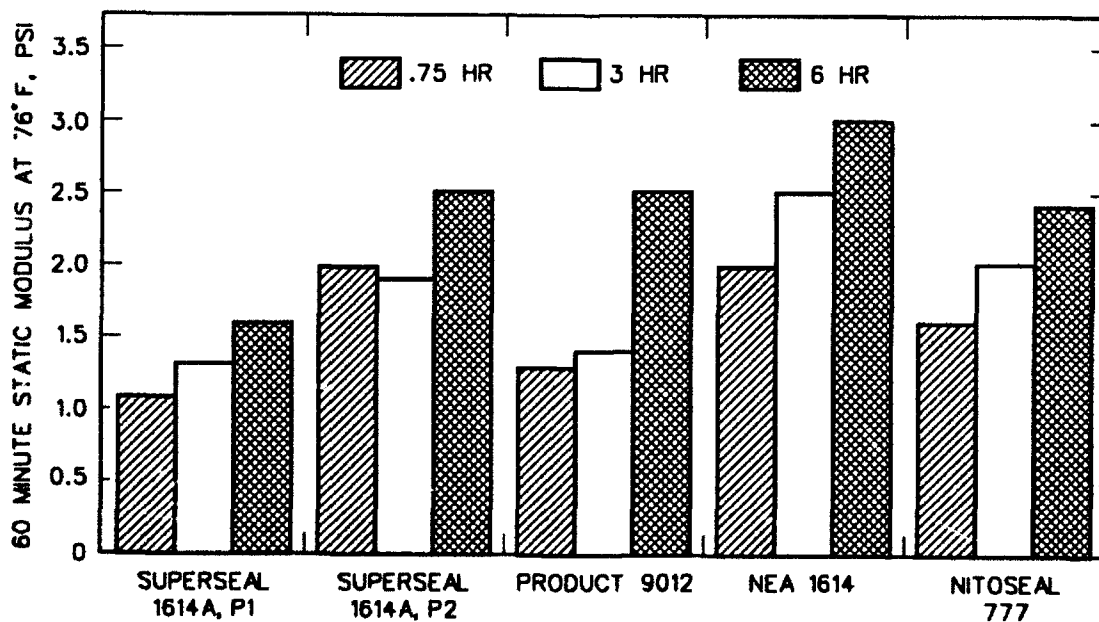


Figure 23. Sixty minute static modulus at 100 percent extension at 76°F for jet-fuel-resistant sealants after 0.75, 3, and 6 hr of heating

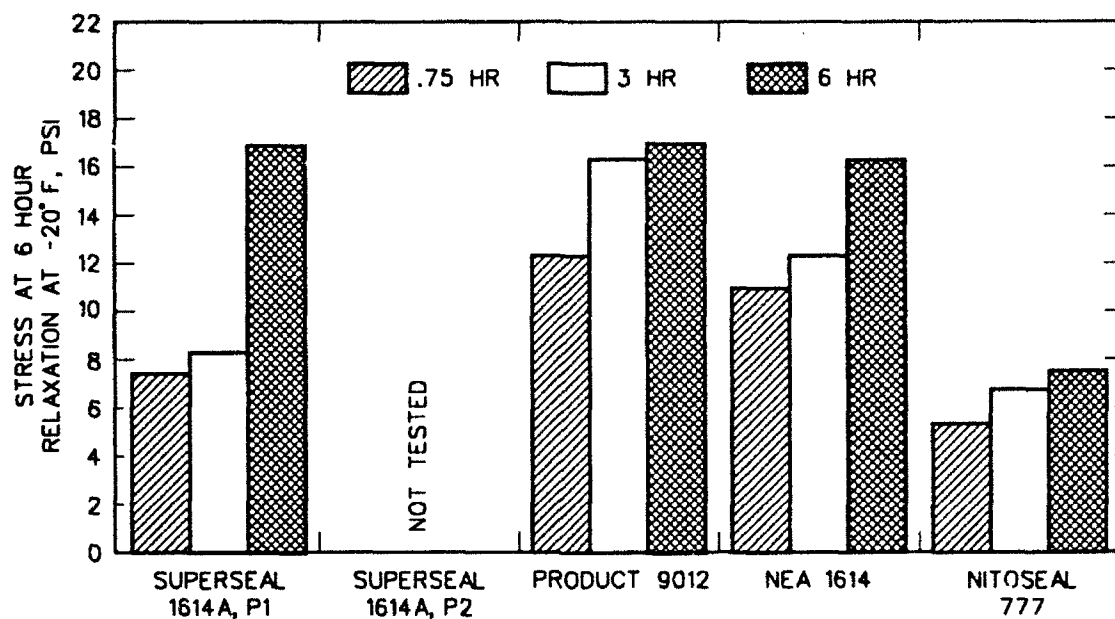


Figure 24. Stress at 6 hr relaxation at -20°F for jet-fuel-resistant sealants after 0.75, 3, and 6 hr of heating

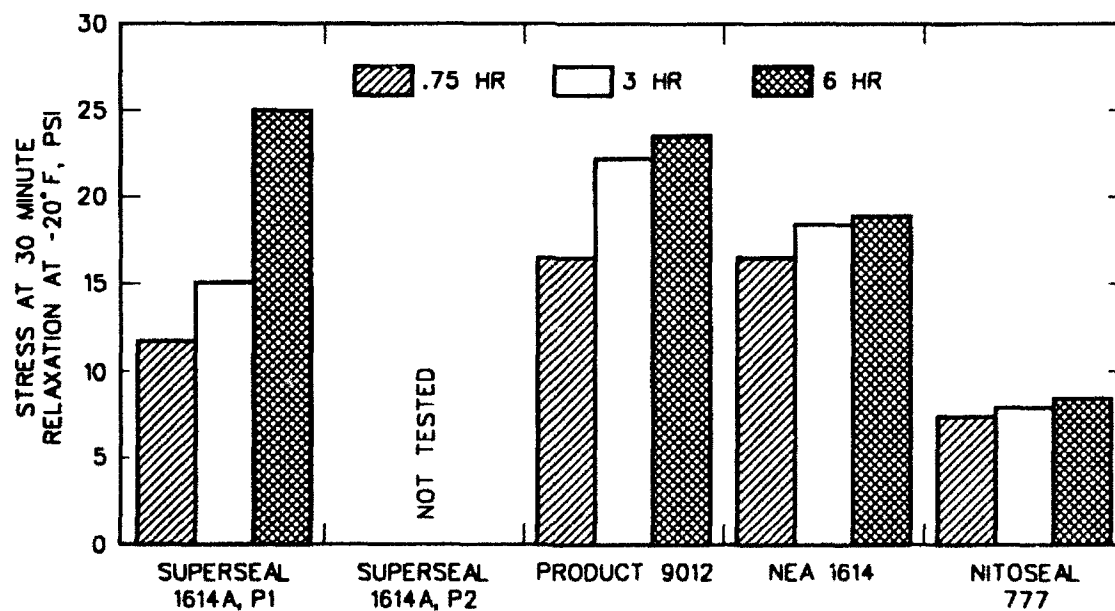


Figure 25. Stress at 30 min relaxation at -20°F for jet-fuel-resistant sealants after 0.75, 3, and 6 hr of heating

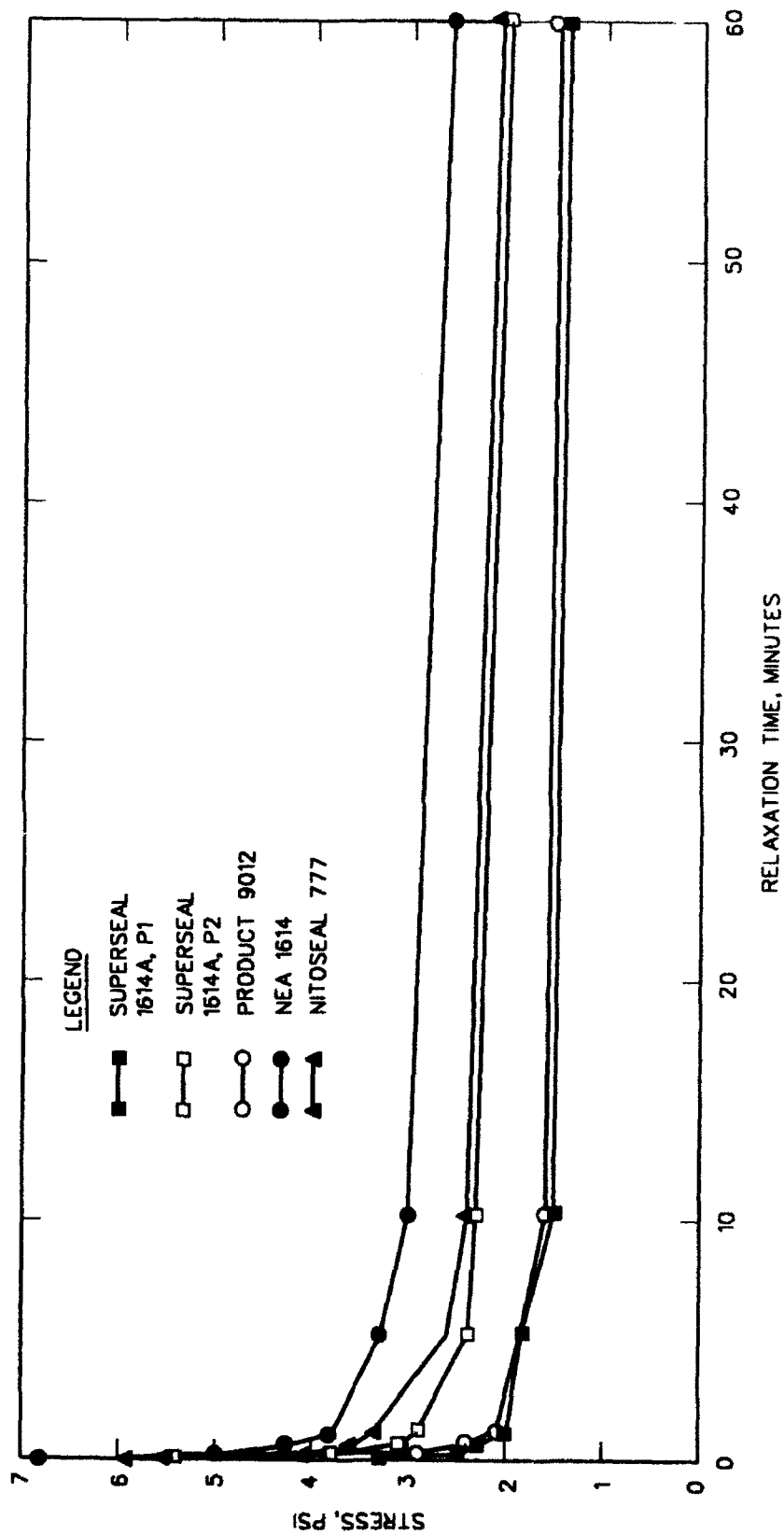


Figure 26. Stress relaxation plots at 76°F for jet-fuel-resistant sealants after 3 hr of heating

Superseal 1614A, Plant 1 after 45 min of heating had the lowest 60 min modulus (1.1 psi), and NEA 1614 after 6 hr of heating had the highest (3.0 psi). Overall, Superseal 1614A, Plant 1 had the lowest modulus values at 76°F. During heating, Nitoseal 777 had the lowest percentage increase in initial static modulus (32 percent) and Superseal 1614A, Plant 2 and NEA 1614 had the highest (78.6 percent and 77.7 percent, respectively). For 60 min relaxed modulus, Product 9012 experienced the greatest increase during heating (92.3 percent) and Superseal 1614A, Plant 2 experienced the lowest (25 percent). Static modulus data at 76°F show that the different sealants tested have stress relaxation characteristics which differ with relaxation period and heating time used to prepare specimens.

53. Figure 27 is a plot of relaxation data at -20°F for the JFR sealants after 3 hr of heating. The stiffness of the sealants prevented them from being extended 100 percent at -20°F; therefore, they were loaded to a 50-psi tensile stress, and relaxation was monitored from that initial stress. Figure 24 shows stress after a 30 min relaxation period at -20°F. As seen from the data, each sealant experienced increased stress, with Nitoseal 777 having the lowest average stress (8.2 psi) and Product 9012 having the highest average stress (20.9 psi). Nitoseal 777 had the lowest stress increase due to heating (11.5 percent), and Superseal 1614A, Plant 1 had the largest (110 percent). Figure 25 shows the 6 hr relaxed stress at -20°F. The data indicate that Nitoseal 777 had the lowest average stress (6.6 psi) and Product 9012 had the highest (15.2 psi). Results show that the sealants have different stress relaxation characteristics at -20°F and different heating period affects the sealants differently.

54. The additional test results indicate that current hot-applied JFR sealants manufactured to meet FS SS-S-1614A are being evaluated at or near their limit of low temperature extension performance at 0°F. Each sealant tested resisted flow at 158°F and some resisted flow at 200°F. All sealants showed resilience characteristics that varied depending on curing and aging periods. These results suggest that more stringent specification parameters of flow and resilience could be incorporated in FS SS-S-1614A to more accurately define physical characteristics of current materials. Stricter bond testing (such as at -20°F), however, would not be possible with current sealants since they could not perform at lower temperatures or higher extensions.

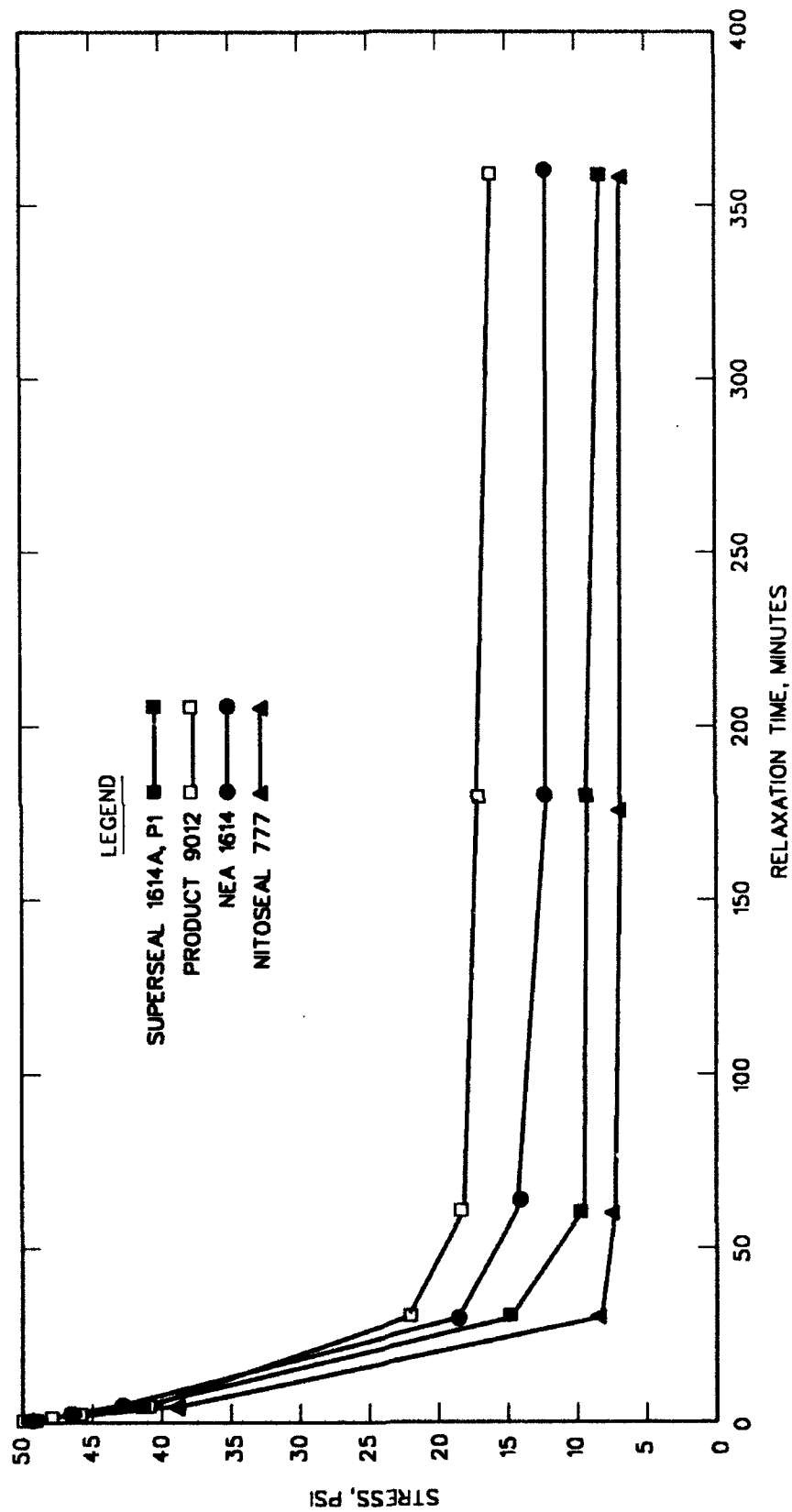


Figure 27. Stress relaxation plots at -20°F for jet-fuel-resistant sealants after 3 hr of heating

PART IV: PHYSICAL CHARACTERISTICS OF AVAILABLE HOT-APPLIED
NON-JET-FUEL-RESISTANT SEALANTS

Products Tested

55. During initial project planning meetings, it was decided that in order to determine specification limits for an improved non-JFR sealant, a variety of available hot-applied, non-JFR sealants should be tested to determine their physical properties. It was also decided that tests in addition to those in FS SS-S-1401C should be conducted. The additional tests would be more extensive and would be conducted to determine performance limits and provide a basis for comparison and improvement. Sealant materials manufactured to meet a variety of specifications including FS SS-S-1401C, ASTM D3405, American Association of State Highway and Transportation Officials (AASHTO) M173, (AASHTO 1986), and modifications of ASTM D3405 and AASHTO M173 were procured for testing. All of those sealants were supplied as solid materials.

56. Joint sealant materials which were evaluated for the non-JFR portion of this study were as follows:

- a. RoadSaver 222, Crafco Inc., manufactured to meet FS SS-S-1401C, ASTM D3405, and ASTM D1190.
- b. RoadSaver 231, Crafco Inc., manufactured to meet several state modified low-modulus ASTM D3405 specifications including Iowa Department of Transportation (IDOT) 4136.02a (IDOT 1984), Kansas Department of Transportation (KDOT) TS109.6 (KDOT 1986), and South Dakota Department of Transportation (SDDOT) low modulus D3405 (SDDOT, undated).
- c. RoadSaver 299, Crafco Inc., manufactured to meet a low modulus company developed specification (Crafco 1991).
- d. RoadSaver 515, Crafco Inc., manufactured to meet a variety of state modified AASHTO M173 specifications such as Missouri 1051.1.4 (Missouri Highway and Transportation Commission 1988), Minnesota Department of Transportation (MNDOT) 3723 (MNDOT 1988), and South Dakota 870.1 (SDDOT 1985).
- e. Product 9030, Koch Materials Company, manufactured to meet several state low-modulus modified ASTM D3405 specifications.
- f. Product 9005, Koch Materials Company, manufactured to meet requirements of FS SS-S-1401C, ASTM D3405, and ASTM D1190.
- g. Sof Seal, W. R. Meadows, manufactured to meet several state low-modulus modified ASTM D3405 specifications.
- h. Hi-Spec, W. R. Meadows, manufactured to meet requirements of FS SS-S-1401C and ASTM D3405.

57. Production lot numbers and the manufacturer's recommended pour temperature and safe heating temperature are as follows:

<u>Product</u>	<u>Lot Identification</u>	<u>Pour Temperature</u>	<u>Safe Heating Temperature</u>
RoadSaver 222	011290T	380°F	410°F
RoadSaver 231	021240E	380°F	410°F
RoadSaver 299	121489F	380°F	410°F
RoadSaver 515	0630890	380°F	410°F
Product 9030	213	370°F	390°F
Product 9005	717	370°F	390°F
Sof Seal	9082	370°F	390°F
Hi-Spec	8210	370°F	390°F

Specification Testing

58. Each of the eight sealant materials was tested in accordance with FS SS-S-1401C using the manufacturer's recommended safe heating temperature. The sealants were also tested for FS SS-S-1401C properties at several other heating periods to determine if the heating period affected measured physical properties. The additional heating periods included 45 min to provide an indication of short term heating and application, and 6 hr to determine the effects of long term heating. Testing for conformance with FS SS-S-1401C at the standard heat period was performed by both WES and CrafcO to determine testing variability between laboratories.

59. WES and CrafcO results are presented in Table 17, and a data summary which provides mean values, estimated standard deviation (based on a value of 0.886 times the range of the two values) (Burr 1974), and coefficient of variation is shown in Table 18. CrafcO results for the non-JFR sealants using heating times of 45 min and 6 hr are summarized in Tables 19 and 20. Data with numerical values are plotted in Figures 28 through 33.

Specification Test Analysis

60. The test results provided by both WES and CrafcO in Table 17 indicate that the RoadSaver 222 and the Hi-Spec sealants conformed to the specification requirements of FS SS-S-1401C. Product 9005 did not meet the unaged resilience initial indentation in the WES testing, but it passed the CrafcO

Table 17

WES and Crafcro Results for Non-Jet-Fuel Resistant SealantsWhen Tested for Conformance with FS SS-S-1401C

FS SS-S-1401C Parameter	RoadSaver 222		RoadSaver 231		RoadSaver 299		RoadSaver 515		FS SS-S-1401C Requirements
	WES	Crafcro	WES	Crafcro	WES	Crafcro	WES	Crafcro	
Penetration, cm	0.73	0.84	1.11	1.29	1.54	1.52	0.76	0.75	0.90 max
Flow, cm	0.0	0.0	0.0	0.0	0.4	0.0	0.0	0.0	0.3 max
Resilience, percent	67	68	47	54	34	42	45	49	60 min
Indentation, cm	0.15	0.13	0.23	0.19	0.34	0.39	0.15	0.11	0.05-0.15
Aged resilience, percent	67	62	48	53	37	43	45	49	60 min
Indentation, cm	0.15	0.12	0.25	0.20	0.34	0.41	0.16	0.12	0.05-0.15
Bond,									
Nonimmersed	S*	S	S	S	S	S	S	S	S
Water immersed	S	S	S	S	S	S	S	S	S
Compatibility	S	S	S	S	S	S	S	S	S

(Continued)

* S signifies satisfactory test results.

Table 17 (Concluded)

FS SS-S-1401C Parameter	Product 9030		Product 9005		Sof-Seal		Hi-Spec		FS SS-S-1401C Requirements
	WFS	Grafco	WES	Grafco	WES	Grafco	WES	Grafco	
Penetration, cm	1.01	1.11	.81	.92	1.24	1.17	0.72	0.65	0.90 max
Flow, cm	0.0	0.0	0.1	0.2	0.0	0.2	0.0	0.1	0.3 max
Resilience, percent	88	90	68	69	67	68	67	61	60 min
Indentation, cm	0.20	0.18	0.17	0.15	0.27	0.21	0.15	0.07	0.05-0.15
Aged resilience, percent	87	89	67	63	77	78	72	70	60 min
Indentation, cm	0.14	0.15	0.15	0.15	0.25	0.11	0.14	0.07	0.05-0.15
Bond,									
Nonimmersed	S	S	S	**	S	S	S	S	S
Water immersed	J†	S	S	**	S	S	S	S	S
Compatibility	S	S	S	S	S	S	S	S	S

** Testing could not be completed due to deformation of testing device grips during extension.

† U signifies unsatisfactory results.

Table 18

Summary of FS SS-S-1401C Data Obtained by WES and Crafcro

FS SS-S-1401C Parameter		RoadSaver 222	RoadSaver 231	RoadSaver 299	RoadSaver 515
Penetration, cm	\bar{X}	0.785	1.20	1.53	0.755
	S	0.097	0.160	0.018	0.009
	CV	12.4 percent	13.3 percent	1.2 percent	1.2 percent
Flow, cm	\bar{X}	0.0	0.0	0.2	0.0
	S	0.0	0.0	0.35	0.0
	CV	0.0 percent	0.0 percent	177.2 percent	0.0
Resilience, percent	\bar{X}	76.5	50.5	38.0	47.0
	S	.89	6.2	7.1	3.5
	CV	1.3 percent	12.3 percent	18.7 percent	7.5 percent
Indentation, cm	\bar{X}	0.14	0.21	0.365	0.13
	S	0.018	0.035	0.044	0.035
	CV	12.7 percent	16.9 percent	12.1 percent	27.3 percent
Aged resilience, percent	\bar{X}	64.5	50.5	40.0	47.0
	S	4.4	4.4	5.3	3.5
	CV	6.9 percent	8.8 percent	13.3 percent	7.5 percent
Age indentation, percent	\bar{X}	0.135	0.225	0.375	0.14
	S	0.027	0.044	0.062	0.035
	CV	19.7 percent	19.7 percent	16.5 percent	25.3 percent

(Continued)

Table 18 (Concluded)

FS SS-S-1401C Parameter	Product 9030	Product 9005	Sof Seal	Hi-Spec	FS SS-S-1401C Sealant Average	Overall Sealant Average
Penetration, cm						
\bar{X}	1.06	0.865	1.205	0.685	0.778	1.011
S	0.089	0.097	0.062	0.062	--	--
CV	8.4%	11.3%	5.1%	9.1%	10.9%	7.8%
Flow, cm						
\bar{X}	0.00	0.15	0.10	0.05	0.067	0.063
S	0.0	0.089	0.177	0.089	--	--
CV	0.0%	59.1%	177.2%	177.2%	78.8%	73.8%
Resilience, percent						
\bar{X}	89.0	68.5	67.5	64.0	66.7	61.5%
S	1.77	0.89	0.89	5.3	--	--
CV	2.0%	1.3%	1.3%	8.3%	3.6%	6.6%
Indentation						
\bar{X}	0.19	0.16	0.24	0.11	0.137	0.193
S	0.018	0.018	0.05	0.071	--	--
CV	9.3%	11.1%	22.2%	64.4%	29.4%	22.0%
Aged resilience						
\bar{X}	88.1	65.0	77.5	71.0	66.8	62.9
S	1.78	3.54	0.89	1.77	--	--
CV	2.0%	5.5%	1.1%	2.5%	5.0%	6.0%
Aged indentation						
\bar{X}	0.145	0.15	0.18	0.105	0.13	0.182
S	0.009	0.0	0.124	0.062	--	--
CV	6.1%	0.0%	68.9%	59.1%	25.3%	26.9%

Notes: \bar{X} - mean

S - standard deviation (calculated as 0.886 times the data range)

CV - coefficient of variation

Table 19

FS SS-S-1401C Test Results for Non-Jet-Fuel Resistant Sealantsafter 45 min Heating Period

FS SS-S-1401C Parameter	Road- Saver 222	Road- Saver 231	Road- Saver 299	Road- Saver 515	Product 9030	Product 9005	Sof- Seal	Hi- Spec	FS SS-S-1401C Requirements
Penetration, cm	0.74	1.20	1.55	0.76	1.04	0.87	1.28	0.67	0.90 max
Flow, cm	0.00	0.00	0.40	0.00	0.00	0.10	0.20	0.20	0.3 max
Resilience, percent	73	62	24	47	90	72	65	59	60 min
Indentation, cm	0.12	0.15	0.30	0.11	0.13	0.13	0.18	0.09	0.05-0.15
Aged resilience, percent	66	60	40	46	86	67	77	69	60 min
Indentation, cm	0.08	0.23	0.32	0.12	0.15	0.13	0.18	0.07	0.05-0.15
Bond,									
Nonimmersed	S*	S	S	S,	S	**	S	S	S
Water immersed	S	S	S	S	S	**	S	S	S
Compatibility	S	S	S	S	S	S	S	S	S

* S signifies satisfactory test results.

** Test could not be completed due to deformation of the extension devices grips.

Table 20

FS SS-S-1401C Test Results for Non-Jet-Fuel Resistant Sealants
after 6-hr Heating Period

FS SS-S-1401C Parameter	Road- Saver 222	Road- Saver 231	Road- Saver 299	Road- Saver 515	Product 9030	Product 9005	Sof- Seal	Hi- Spec	FS SS-S-1401C Requirements
Penetration, cm	0.83	1.14	1.68	0.69	1.24	0.96	1.14	0.70	0.90 max
Flow, cm	0.00	0.00	0.50	0.00	0.00	0.20	0.10	0.20	0.3 max
Resilience, percent	68	63	45	48	90	64	74	69	60 min
Indentation, cm	0.15	0.13	0.39	0.06	0.19	0.17	0.19	0.13	0.05-0.15
Aged resilience, percent	57	52	52	47	90	64	76	75	60 min
Indentation, cm	0.13	0.18	0.34	0.11	0.20	0.09	0.26	0.10	0.05-0.15
Bond, Nonimmersed	S*	S	S	S	S	**	S	S	S
Water immersed	S	S	S	S	S	**	S	S	S
Compatibility	S	S	S	S	S	S	S	S	S

* S signifies satisfactory test results.

** Test could not be completed due to deformation of the extension devices grips.

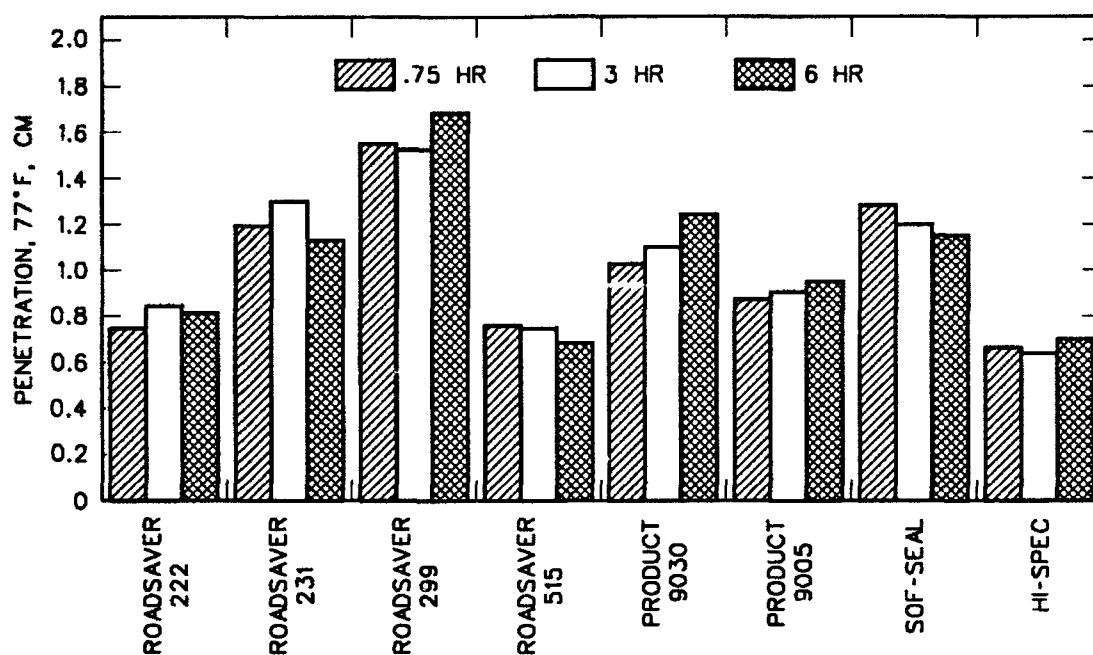


Figure 28. Penetration of non-jet-fuel-resistant sealants after 0.75, 3, and 6 hr of heating

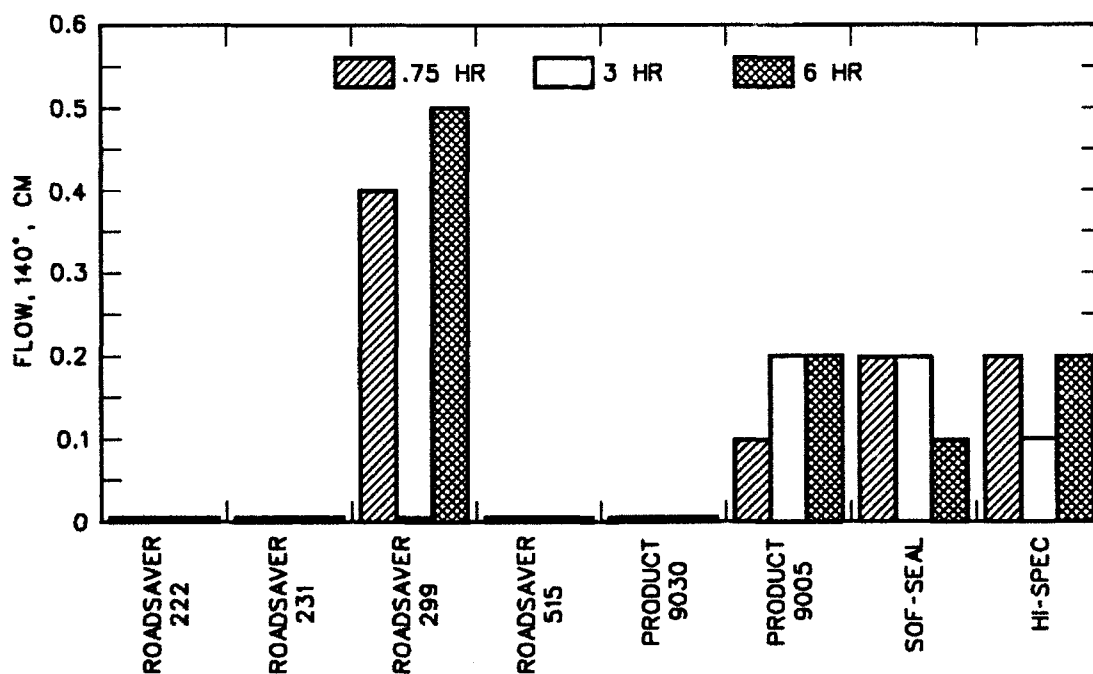


Figure 29. Flow of non-jet-fuel-resistant sealants after 0.75, 3, and 6 hr of heating

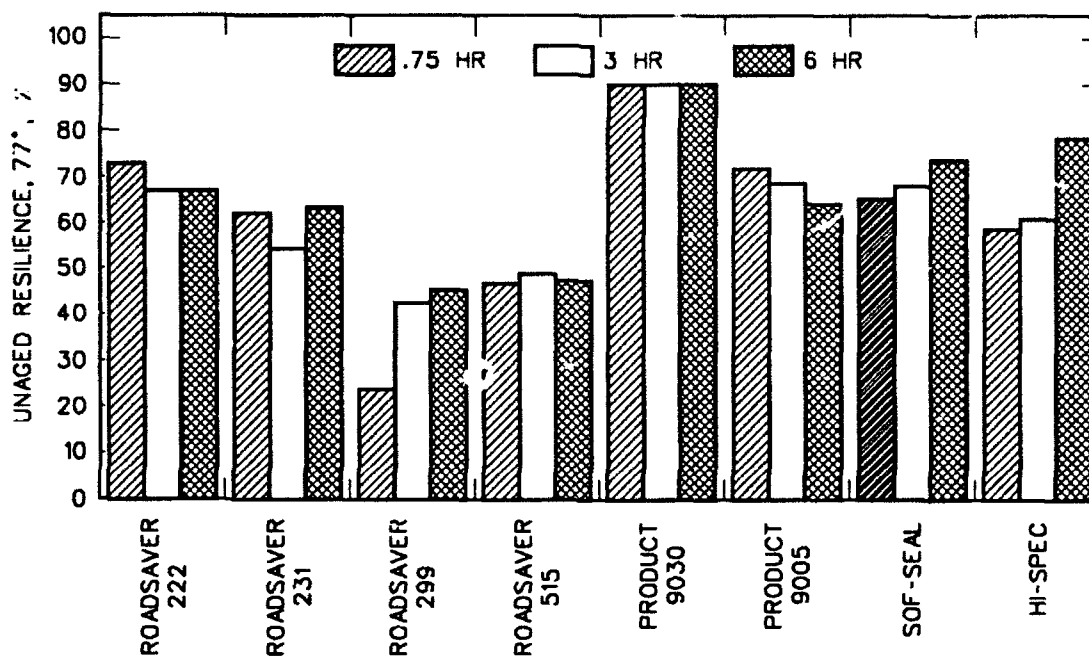


Figure 30. Unaged resilience of non-jet-fuel-resistant sealants after 0.75, 3, and 6 hr of heating

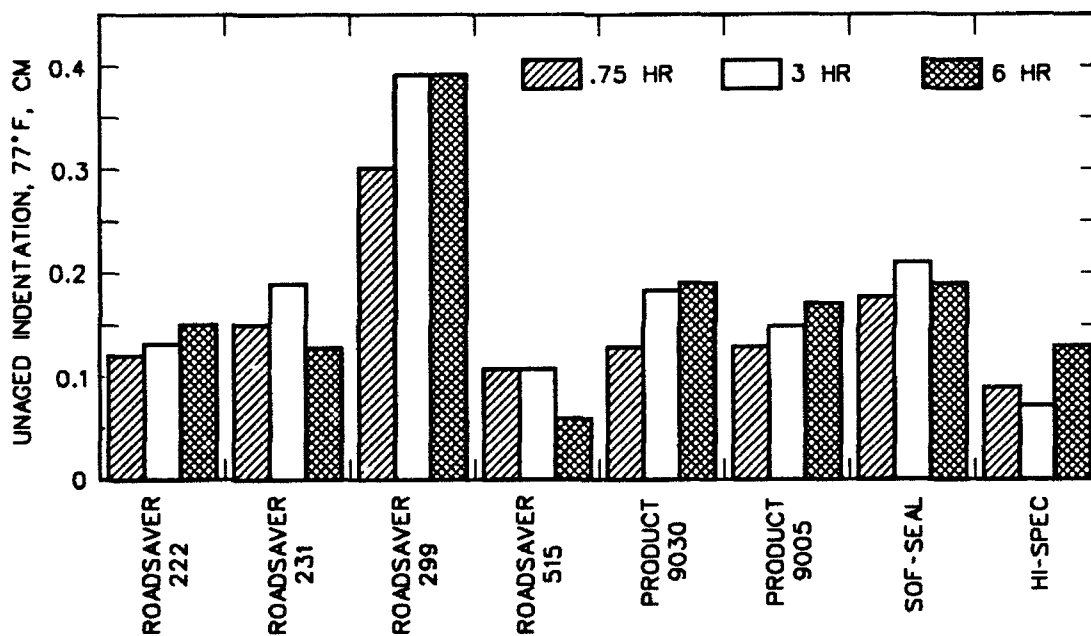


Figure 31. Unaged indentation of non-jet-fuel-resistant sealants after 0.75, 3, and 6 hr of heating

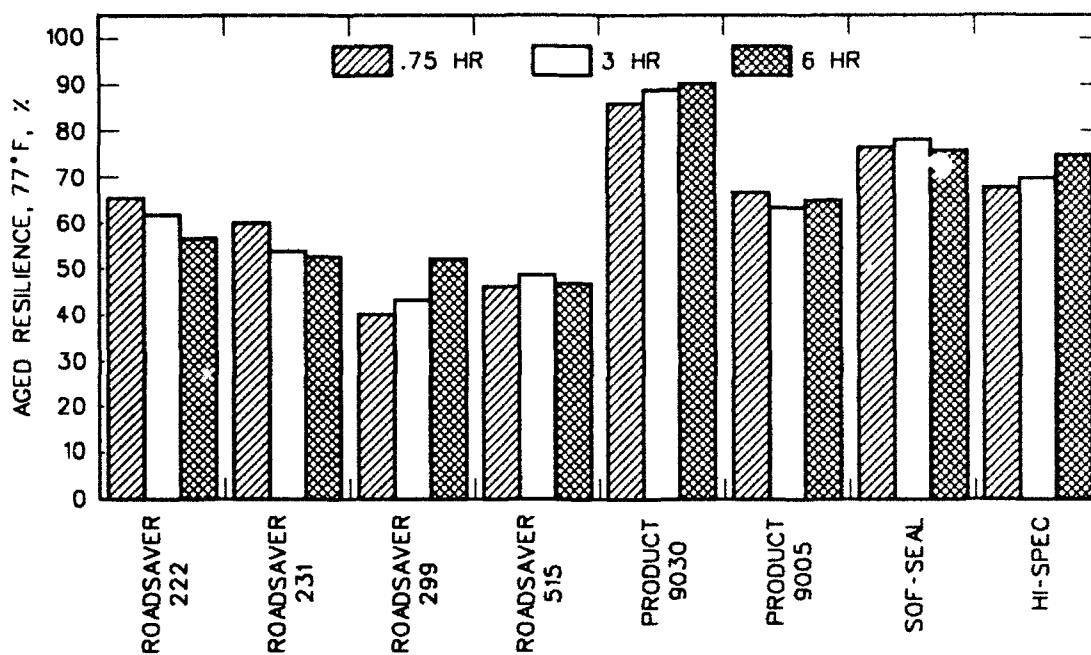


Figure 32. Aged resilience of non-jet-fuel-resistant sealants after 0.75, 3, and 6 hr of heating

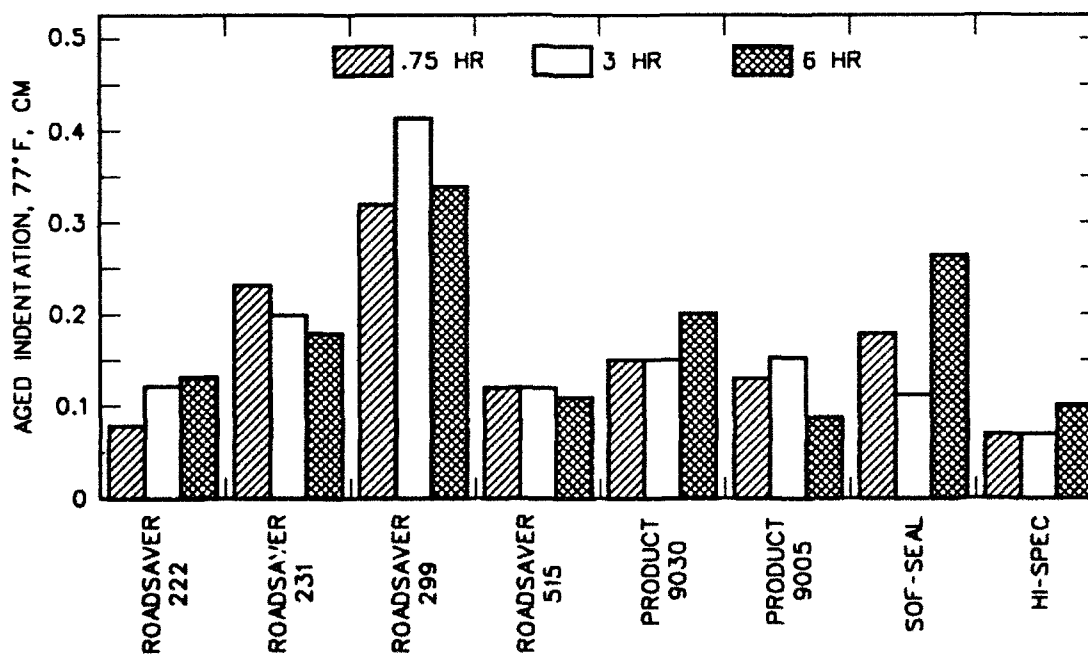


Figure 33. Aged indentation of non-jet-fuel-resistant sealants after 0.75, 3, and 6 hr of heating

testing. Crafco could not conduct the bond testing of the Product 9005 because the samples deformed the specimen grips on the extensiometer.

61. The other non-JFR sealant materials did not conform to the requirements of FS SS-S-1401C in testing conducted by both Crafco and WES.

62. RoadSaver 231, RoadSaver 299, Product 9030, and Sof-Seal sealants were all softer materials as indicated by the penetration and resilience initial indentation results. The RoadSaver 515 material had similar penetration values to typical FS SS-S-1401C materials but it had a lower resilience. It is believed that since these sealants appear to be softer than the SS-S-1401C materials that they may exhibit better low temperature characteristics.

63. The summarized data in Table 18 show variability in testing between the two laboratories. The average penetration values for the sealants range from 0.69 cm to 1.53 cm. The wide range of results was expected since many of the sealants are manufactured to be softer than required by FS SS-S-1401C. The CV for the individual sealants range from 1.2 percent for the RoadSaver 299 and RoadSaver 515 to 13.3 percent for RoadSaver 231. The flow results for the non-JFR sealants show greater variability with the CV as high as 177 percent. The large values of CV were caused by one laboratory's report of no flow while the other laboratory reported flow. The unaged resilience percent recovery CV value ranged from a low of 1.3 percent for the RoadSaver 222 to a high of 18.7 percent for the RoadSaver 299. The results of other test procedures show similar variances in CV as did the penetration, flow, and unaged resilience percent recovery. The variances were significant enough to create the possibility of a sealant sample being tested and reported as conforming to test requirements by one laboratory, but nonconforming to the requirements by a second laboratory.

64. The data obtained on the sealant materials using the additional heating times are provided in Tables 19 and 20 and plotted in Figures 28 through 33.

65. The RoadSaver 222 sample met the specification test requirements when heated for 45 min, but did not meet the aged resilience percent recovery when heated for 6.0 hr. The sealant appeared to soften during heating as indicated by an increase in penetration values. The RoadSaver 222 also showed a decrease in the resilience percent recovery indicating that the sealant experienced a loss in elasticity after aging. The flow and bond test results did not show a change.

66. RoadSaver 231 did not conform with the test requirements of FS SS-S-1401C after both the 45 min and 6 hr heating times. The penetration values indicate the sealant hardens as it is heated. The difference in penetration values between the 3 hr heating time and the 6 hr heating time shows a slight softening but the significance of the change is not clear. Flow and bond test results were not affected by the change in heating times.

67. RoadSaver 299 did not conform to the test requirements of FS SS-S-1401C at either the 45 min or 6-hr heating time. The only area of conformance were the bond and compatibility requirements. The material exhibited a softening as the heating time was increased.

68. RoadSaver 515 did not conform to the FS SS-S-1401C test requirement after 45 min or 6 hr of heating. The penetration values indicated that the material stiffens as the heating time is extended. The other physical properties did not change significantly.

69. Product 9030 did not conform to the FS SS-S-1401C test requirements after the 45 min or 6-hr heating times. The sealant softened with an increase in the penetration values from 1.04 to 1.24 cm. The flow and bond results were not affected by the increase in heating times, and the resilience values did not vary significantly.

70. Product 9005 met the FS SS-S-1401C test requirements after the 45 min heating time but did not meet the penetration and unaged resilience initial indentation after the 6-hr heating time. The material softens with an increase in heating times as indicated by the penetration values.

71. The Sof-Seal material did not conform to the test requirements of FS SS-S-1401C after the 45 min or 6-hr heating time. The material specifically did not meet the penetration or the unaged and aged initial indentation at either heating time. The sealant exhibited stiffening with increased heating times as illustrated by a decrease in penetration values. The flow and bond testing results were not affected by the increased heating time.

72. The Hi-Spec material did not conform to the FS SS-S-1401C test requirements after the 45-min heating time but did meet the requirements after the 6-hr heating time. The values for penetration and resilience varied as heating times changed, but no trends were evident. Bond test results were not affected by changing the heating time.

Additional Laboratory Testing

73. In order to further evaluate the physical characteristics of the non-JFR sealants, additional tests were performed. The additional procedures were selected to identify more clearly the limits of performance of the available non-JFR sealants. Additional tests for sealants meeting other specifications were used to further identify levels of low temperature performance and adhesion. The additional tests were performed on materials heated for 45 min, 3 hr, and 6 hr to determine the effects of various heating periods.

74. Procedures used to determine additional properties were as follows:

- a. Nonimmersed bond. Nonimmersed bond testing was performed as required by FS SS-S-1401C except the specimens were extended 100 percent or 200 percent at -20°F. These higher extensions were performed to determine the limits of low temperature extension capabilities.
- b. Water immersed bond. Water immersed bond testing was performed as required by FS SS-S-1401C except that specimens were extended 100 percent or 200 percent at -20°F.
- c. Tensile adhesion. Tensile adhesion testing was performed using the ASTM D3583 procedure. Testing was performed to determine the adhesion and extension characteristics of the sealants at 77°F.
- d. Softening point. The softening point of the sealants was evaluated according to ASTM D36 to determine the limits of high temperature performance on the sealants.
- e. Ductility. Ductility at 77°F and 39.2°F was determined using an extension rate of 5 cm/min in accordance with ASTM D113 procedure. The test provides an indication of extension characteristics of the sealants.
- f. Penetration at 0°F. Penetration testing was performed at 0°F to provide an indication of low temperature sealant stiffness. Testing was performed using the apparatus and procedures specified in FS SS-S-1401C except that the specimen was conditioned at $0 \pm 2^\circ\text{F}$ for 16 ± 4 hr in a freezer immediately after determining the 77°F penetration. Testing consisted of removing the specimen from the 0°F atmosphere and testing immediately at standard laboratory conditions for penetration with the 150-g cone using a 5-sec duration. These determinations were made within 60 sec from the time of removal from the 0°F atmosphere. The results of the three determinations were averaged and reported.
- g. Viscosity. The heated sealant was tested at the safe heating temperature ($\pm 2^\circ\text{F}$) immediately before pouring test samples. Testing was performed using a Brookfield Model HAT viscometer with appropriate probes and speeds (typically probe 3 at 20 rpm).

- h. Static adhesion. Static adhesion testing was performed to further evaluate adhesion characteristics of the sealants. Testing consisted of (1) preparing standard bond test specimens in accordance with FS SS-S-1401C, (2) conditioning the specimens at 0°F for a minimum of 4 hr, (3) extending the specimens at 0°F to 100 percent extension using the extension rate specified in FS SS-S-1401C, and (4) removing the extended specimens from the freezer and inserting 1-in.-thick spacers to maintain the 100 percent extension. Specimens were then stored at laboratory standard conditions for 24 hr and then examined for any loss of adhesion to the concrete blocks. A passing result was one in which there was less than 160 mm² of adhesive separation. A failing result was a test in which there was more than 160 mm² of adhesive separation.
- i. Stress relaxation. Stress relaxation testing was performed on the sealants after each heating period at both 76 ± 1°F and -20°F ± 2°F. The procedure was the same as the procedure described in Paragraph 33i.

75. Additional physical test data (excluding stress relaxation) are summarized in Tables 21 through 28. Appropriate data are plotted in Figures 34 through 39. Stress relaxation results are summarized in Tables 29 through 36, and data are plotted in Figures 40 through 44.

Additional Laboratory Test Analysis

76. Satisfactory results for all sealants tested after each heating period were recorded for the nonimmersed bond testing at -20°F using 100 percent extension. Product 9005 could not be tested because the extension device was damaged during the test. Testing at -20°F using 200 percent extension revealed unsatisfactory results for RoadSaver 222, RoadSaver 515, and Hi-Spec sealants. Satisfactory results were obtained for RoadSaver 231, RoadSaver 299, Product 9030, and Sof-Seal.

77. Water immersed bond test results were the same as for nonimmersed bond testing, except for the RoadSaver 222 after 6 hr of heating and Product 9030 after 45 min of heating. The test results of these sealants were unsatisfactory. These bond results show that the FS SS-S-1401C materials tested can withstand a greater amount of extension (up to 100 percent) than the 50 percent requirement provided in the specification. However, the FS SS-S-1401C materials failed when tested using 200 percent extension. The low-modulus sealants tested can satisfactorily withstand 200 percent elongation; therefore, they may have performance advantages at colder temperatures.

Table 21
Additional Physical Characteristics
Crafco 222

Test Parameter	Heating Period		
	45 min	3 hr	6 hr
Bond, nonimmersed			
-20°F, 100 percent, 3 cycles	S*	S	S
-20°F, 200 percent, 3 cycles	U**	U	U
Bond, water immersed			
-20°F, 100 percent, 3 cycles	S	S	U
-20°F, 200 percent, 3 cycles	U	U	U
Tensile adhesion, percent	617	667	567
Softening point, °F	184	182	185
Ductility, 77°F, 5 cm/min, cm	45	53	45
Ductility, 39.2°F 5 cm/min, cm	62	61	68
Penetration, 0°F, cm	0.15	0.18	0.17
Viscosity, Brookfield, 410°F, cps	3,640	3,300	3,750
Static adhesion	Pass	Pass	Pass

* S signifies satisfactory performance in the bond test using FS SS-S-1401C criteria.

** U signifies unsatisfactory performance.

Table 22
Additional Physical Characteristics
Crafco 231

Test Parameter	Heating Period		
	45 min	3 hr	6 hr
Bond, nonimmersed			
-20°F, 100 percent, 3 cycles	S*	S	S
-20°F, 200 percent, 3 cycles	S	S	S
Bond, water immersed			
-20°F, 100 percent, 3 cycles	S	S	S
-20°F, 200 percent, 3 cycles	S	S	S
Tensile adhesion, percent	1,083	1,100	1,067
Softening point, °F	216	208	214
Ductility, 77°F, 5 cm/min, cm	73	66	56
Ductility, 39.2°F 5 cm/min, cm	91	84	76
Penetration, 0°F, cm	0.32	0.30	0.30
Viscosity, Brookfield, 410°F, cps	1,700	1,800	1,400
Static adhesion	Pass	Pass	Pass

* S signifies satisfactory performance in the bond test using FS SS-S-1410C criteria.

Table 23
Additional Physical Characteristics
Crafco 299

Test Parameter	Heating Period		
	45 min	3 hr	6 hr
Bond, nonimmersed			
-20°F, 100 percent, 3 cycles	S*	S	S
-20°F, 200 percent, 3 cycles	S	S	S
Bond, water immersed			
-20°F, 100 percent, 3 cycles	S	S	S
-20°F, 200 percent, 3 cycles	S	S	S
Tensile adhesion, percent	1,000	933	850
Softening point, °F	185	183	182
Ductility, 77°F, 5 cm/min, cm	48	48	46
Ductility, 39.2°F 5 cm/min, cm	43	52	40
Penetration, 0°F, cm	0.42	0.50	0.54
Viscosity, Brookfield, 410°F, cps	2,600	1,960	1,600
Static adhesion	Pass	Pass	Pass

* S signifies satisfactory performance in the bond test using FS SS-S-1410C criteria.

Table 24
Additional Physical Characteristics
Crafco 515

Test Parameter	Heating Period		
	45 min	3 hr	6 hr
Bond, nonimmersed			
-20°F, 100 percent, 3 cycles	S*	S	S
-20°F, 200 percent, 3 cycles	U**	U	U
Bond, water immersed			
-20°F, 100 percent, 3 cycles	S	S	S
-20°F, 200 percent, 3 cycles	U	U	U
Tensile adhesion, percent	916	883	900
Softening point, °F	205	202	195
Ductility, 77°F, 5 cm/min, cm	35	35	36
Ductility, 39.2°F 5 cm/min, cm	30	35	34
Penetration, 0°F, cm	0.16	0.17	0.17
Viscosity, Brookfield, 410°F, cps	3,680	3,680	2,500
Static adhesion	Pass	Pass	Pass

* S signifies satisfactory performance in the bond test using FS
SS-S-1410C criteria.

** U signifies unsatisfactory performance.

Table 25
Additional Physical Characteristics
Product 9030

Test Parameter	Heating Period		
	45 min	3 hr	6 hr
Bond, nonimmersed			
-20°F, 100 percent, 3 cycles	S*	S	S
-20°F, 200 percent, 3 cycles	S	S	S
Bond, water immersed			
-20°F, 100 percent, 3 cycles	S	S	S
-20°F, 200 percent, 3 cycles	U**	S	S
Tensile adhesion, percent	700	650	550
Softening point, °F	191	187	180
Ductility, 77°F, 5 cm/min, cm	23A†	32A	29A
Ductility, 39.2°F 5 cm/min, cm	43	44	48
Penetration, 0°F, cm	0.46	0.47	0.51
Viscosity, Brookfield, 390°F, cps	1,000	1,150	800
Static adhesion	Fail	Fail	Fail

* S signifies satisfactory performance in the bond test using FS SS-S-1410C evaluation criteria.

** U signifies unsatisfactory performance.

† A signifies adhesive separation during the test procedure.

Table 26
Additional Physical Characteristics
Product 9005

<u>Test Parameter</u>	<u>Heating Period</u>		
	<u>45 min</u>	<u>3 hr</u>	<u>6 hr</u>
Bond, nonimmersed			
-20°F, 100 percent, 3 cycles	*	*	*
-20°F, 200 percent, 3 cycles	*	*	*
Bond, water immersed			
-20°F, 100 percent, 3 cycles	*	*	*
-20°F, 200 percent, 3 cycles	*	*	*
Tensile adhesion, percent	883	750	808
Softening point, °F	184	183	183
Ductility, 77°F, 5 cm/min, cm	37	35	34
Ductility, 39.2°F 5 cm/min, cm	49	48	47
Penetration, 0°F, cm	0.08	0.07	0.10
Viscosity, Brookfield, 390°F, cps	800	700	700
Static adhesion	Pass	Pass	Pass

* The sealant stiffness caused bent grips in the extension machine; therefore, bond test could not be completed.

Table 27
Additional Physical Characteristics
Sof-Seal

<u>Test Parameter</u>	<u>Heating Period</u>		
	<u>45 min</u>	<u>3 hr</u>	<u>6 hr</u>
Bond, nonimmersed			
-20°F, 100 percent, 3 cycles	S*	S	S
-20°F, 200 percent, 3 cycles	S	S	S
Bond, water immersed			
-20°F, 100 percent, 3 cycles	S	S	S
-20°F, 200 percent, 3 cycles	S	S	S
Tensile adhesion, percent**	1,033	900	716
Softening point, °F	170	186	184
Ductility, 77°F, 5 cm/min, cm	47	52	51
Ductility, 39.2°F 5 cm/min, cm	48	52	49
Penetration, 0°F, cm	0.48	0.47	0.43
Viscosity, Brookfield, 390°F, cps	1,700	2,200	2,200
Static adhesion	Pass	Pass	Pass

* S signifies satisfactory performance in the bond test using FS
SS-S-1410C evaluation criteria.

** Amount of elongation a bond sample can be stretched.

Table 28
Additional Physical Characteristics
Hi-Spec

Test Parameter	Heating Period		
	45 min	3 hr	6 hr
Bond, non-immersed			
-20°F, 100 percent, 3 cycles	S*	S	S
-20°F, 200 percent, 3 cycles	U**	U	U
Bond, water immersed			
-20°F, 100 percent, 3 cycles	S	S	S
-20°F, 200 percent, 3 cycles	U	U	U
Tensile adhesion, percent	916	750	783
Softening point, °F	174	181	196
Ductility, 77°F, 5 cm/min, cm	42	41	57
Ductility, 39.2°F 5 cm/min, cm	39	43	51
Penetration, 0°F, cm	0.15	0.15	0.16
Viscosity, Brookfield, 390°F, cps	2,900	2,880	3,200
Static adhesion	Pass	Pass	Pass

* S signifies satisfactory performance in the bond test using FS
SS-S-1410C criteria.

** U signifies unsatisfactory performance.

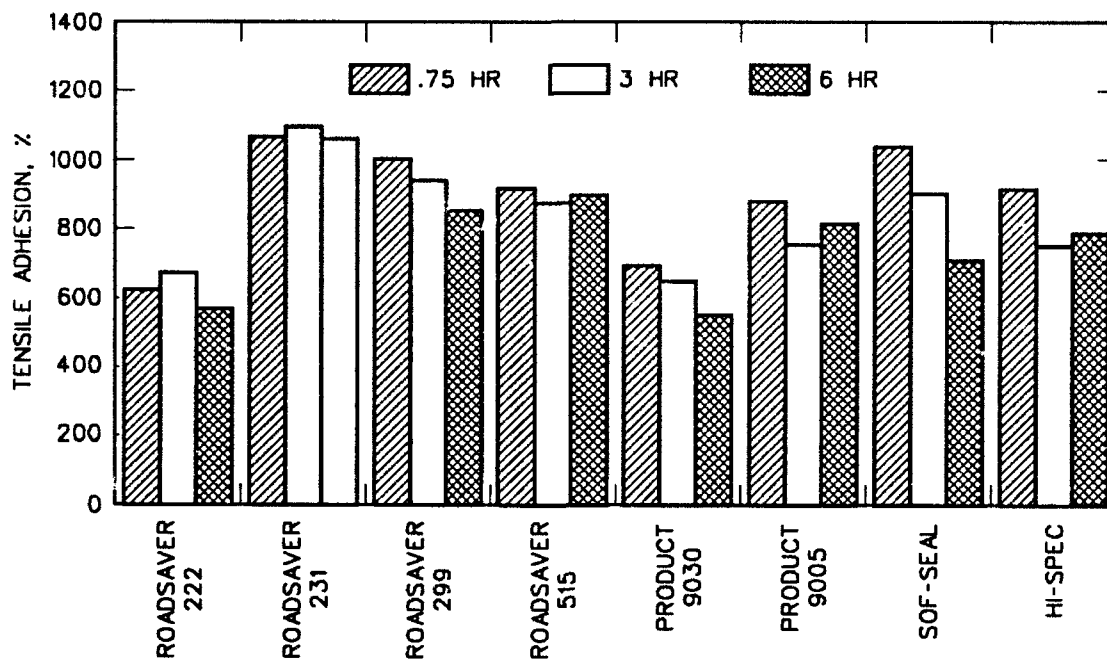


Figure 34. Tensile adhesion of non-jet-fuel-resistant sealants after 0.75, 3, and 6 hr of heating

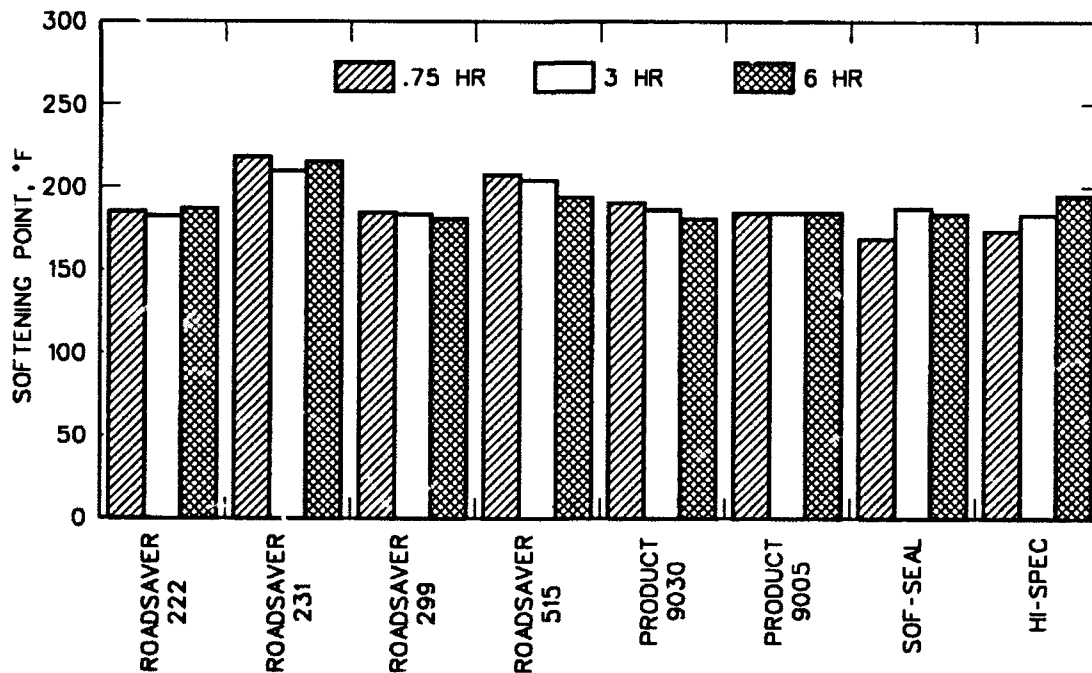


Figure 35. Softening point of non-jet-fuel-resistant sealants after 0.75, 3, and 6 hr of heating

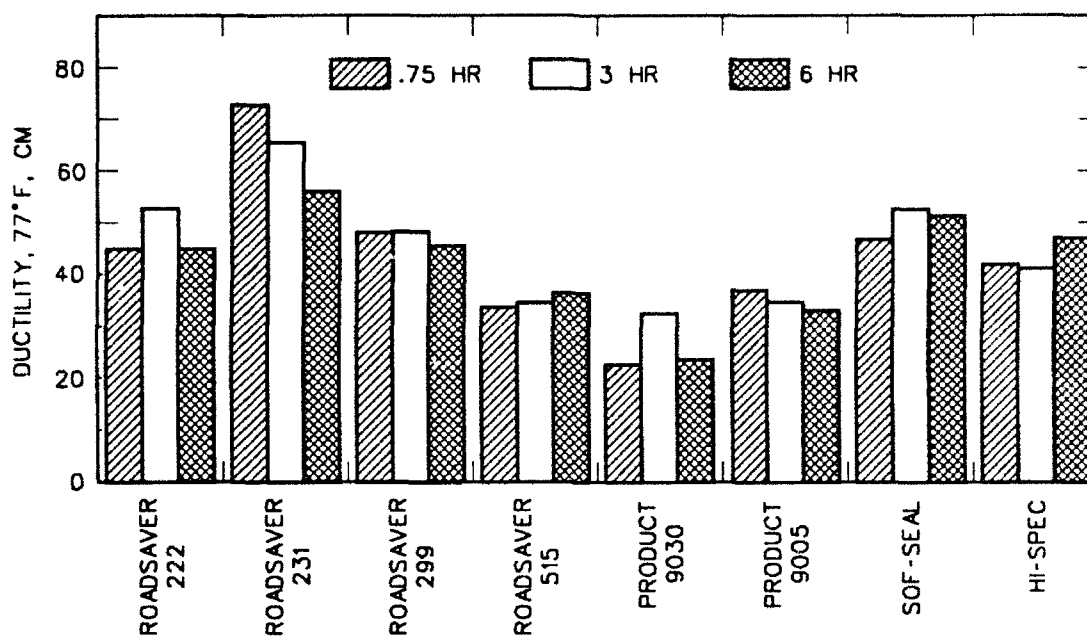


Figure 36. Ductility at 77°F of non-jet-fuel-resistant sealants after 0.75, 3, and 6 hr of heating

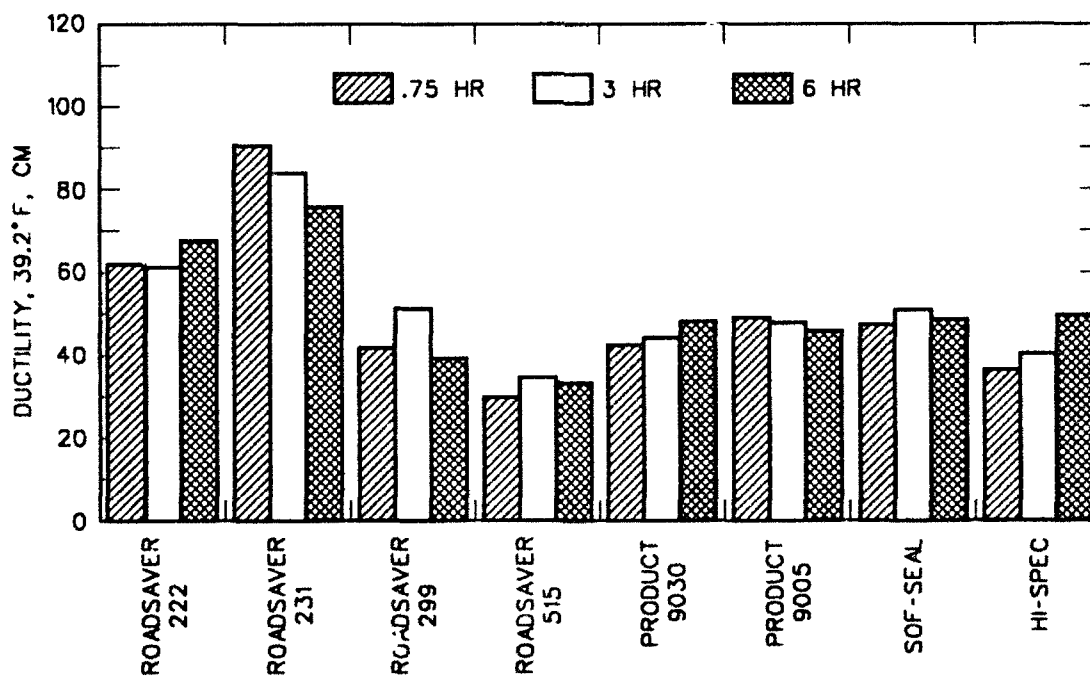


Figure 37. Ductility at 39.2°F of non-jet-fuel-resistant sealants after 0.75, 3, and 6 hr of heating

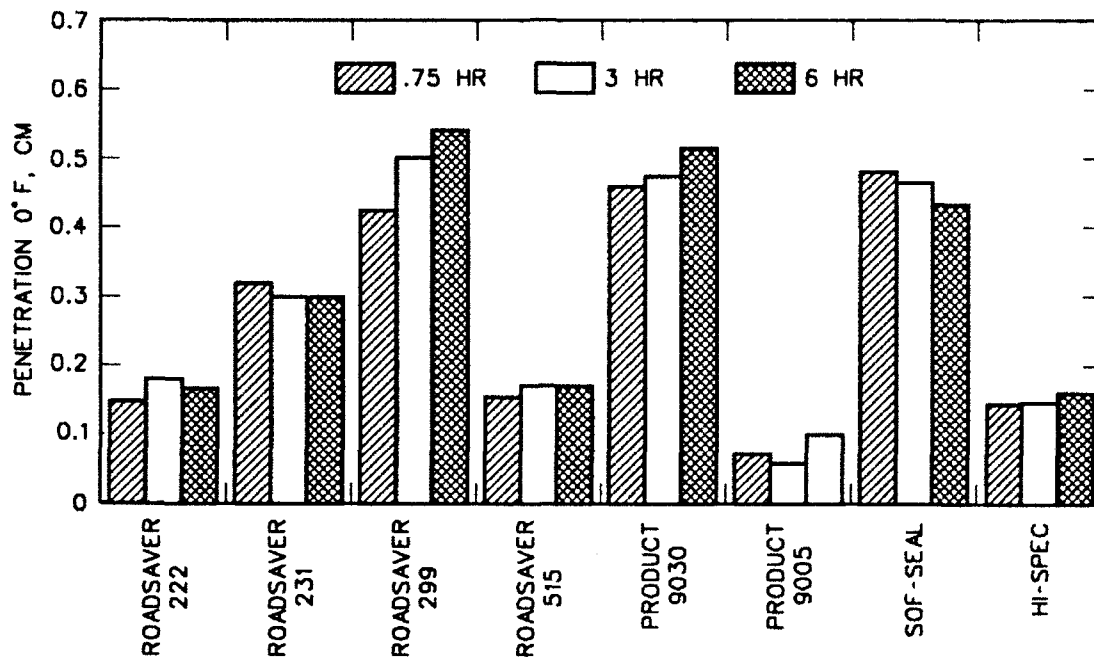


Figure 38. Penetration at 0°F of non-jet-fuel-resistant sealants after 0.75, 3, and 6 hr of heating

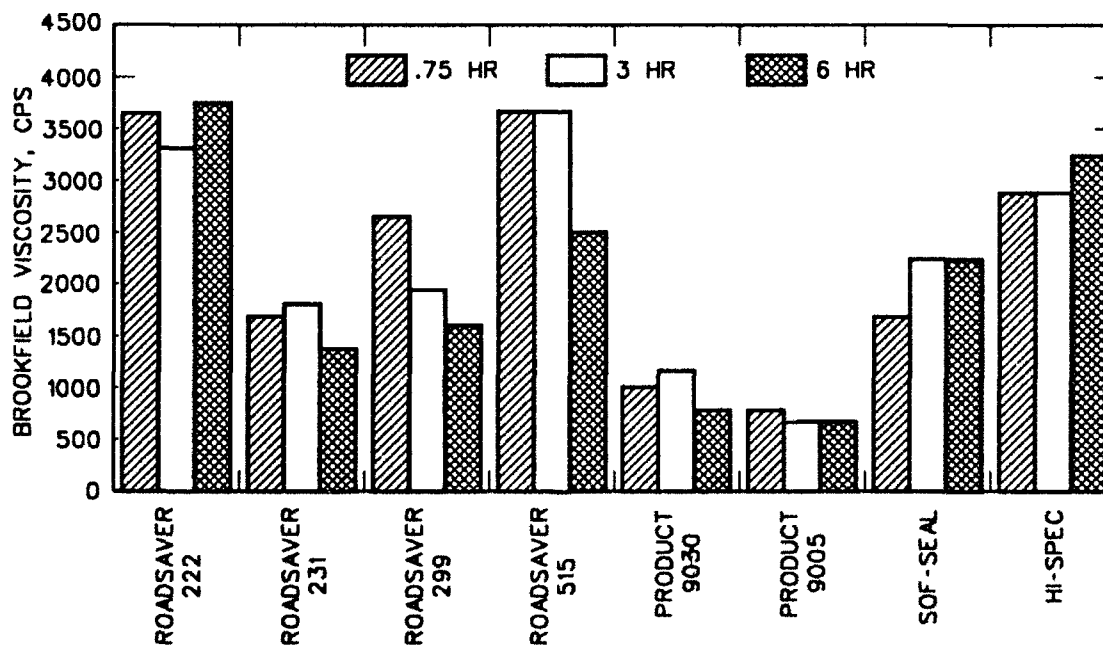


Figure 39. Viscosity at safe heating temperature of non-jet-fuel-resistant sealants after 0.75, 3, and 6 hr of heating

Table 29
Stress Relaxation Data at $76 \pm 1^\circ\text{F}$ and $-20 \pm 2^\circ\text{F}$
RoadSaver 222

<u>Relaxation Period</u>	<u>Stress, psi</u>		
	<u>45 min*</u>	<u>3.0 hr</u>	<u>6.0 hr</u>
<u>Testing at $76 \pm 1^\circ\text{F}$</u>			
0 sec	6.0	5.3	4.8
10 sec	4.8	3.8	3.5
30 sec	4.0	3.3	3.0
60 sec	3.8	3.0	2.8
5 min	2.8	2.3	2.3
10 min	2.6	2.1	2.0
60 min	2.3	1.6	1.6
<u>Testing at $-20 \pm 2^\circ\text{F}$</u>			
0 sec	50.0	50.0	50.0
30 sec	46.3	47.9	46.5
60 sec	43.5	45.6	43.3
2 min	39.5	41.1	38.3
5 min	30.1	30.1	28.7
30 min	15.5	10.0	13.7
60 min	11.4	9.1	9.1
3 hr	11.0	8.7	9.1
6 hr	10.3	8.7	8.7

* Heating period used to prepare specimens.

Table 30
Stress Relaxation Data at $76 \pm 1^\circ\text{F}$ and $-20 \pm 2^\circ\text{F}$
RoadSaver 231

<u>Relaxation Period</u>	<u>Stress, psi</u>		
	<u>45 min*</u>	<u>3.0 hr</u>	<u>6.0 hr</u>
<u>Testing at $76 \pm 1^\circ\text{F}$</u>			
0 sec	2.5	2.8	3.0
10 sec	1.8	2.0	2.0
30 sec	1.3	1.8	1.5
60 sec	1.2	1.5	1.5
5 min	1.1	1.1	1.0
10 min	0.8	1.0	1.0
60 min	0.6	0.6	0.8
<u>Testing at $-20 \pm 2^\circ\text{F}$</u>			
0 sec	50.0	50.0	50.0
30 sec	42.4	40.1	42.0
60 sec	35.5	33.3	34.5
2 min	31.0	28.3	27.2
5 min	20.5	18.7	17.1
30 min	4.6	5.2	4.4
60 min	4.5	5.0	4.4
3 hr	4.1	5.0	3.9
6 hr	4.1	4.5	3.9

* Signifies heating period used to prepare specimens.

Table 31
Stress Relaxation Data at $76 \pm 1^\circ\text{F}$ and $-20 \pm 2^\circ\text{F}$
RoadSaver 299

<u>Relaxation Period</u>	<u>Stress, psi</u>		
	<u>45 min*</u>	<u>3.0 hr</u>	<u>6.0 hr</u>
<u>Testing at $76 \pm 1^\circ\text{F}$</u>			
0 sec	1.2	1.3	1.6
10 sec	0.8	1.0	1.0
30 sec	0.5	0.8	1.0
60 sec	0.5	0.8	0.8
5 min	0.4	0.5	0.5
10 min	0.3	0.5	0.5
60 min	0.3	0.3	0.3
<u>Testing at $-20 \pm 2^\circ\text{F}$</u>			
0 sec	50.0	50.0	50.0
30 sec	25.1	20.5	19.1
60 sec	17.8	14.2	13.3
2 min	5.7	9.1	8.3
5 min	5.0	3.7	2.8
30 min	1.4	2.3	2.3
60 min	0.9	1.8	1.4
3 hr	0.5	0.9	0.9
6 hr	0.5	0.5	0.9

* Heating period used to prepare specimens.

Table 32
Stress Relaxation Data at $76 \pm 1^\circ\text{F}$ and $-20 \pm 2^\circ\text{F}$
RoadSaver 515

<u>Relaxation Period</u>	<u>Stress, psi</u>		
	<u>45 min*</u>	<u>3.0 hr</u>	<u>6.0 hr</u>
<u>Testing at $76 \pm 1^\circ\text{F}$</u>			
0 sec	3.3	3.5	3.8
10 sec	2.0	2.4	3.0
30 sec	1.6	1.9	2.0
60 sec	1.5	1.6	1.9
5 min	1.0	1.0	1.4
10 min	1.0	1.0	1.0
60 min	0.5	0.5	0.5
<u>Testing at $-20 \pm 2^\circ\text{F}$</u>			
0 sec	50.0	50.0	50.0
30 sec	45.2	47.9	46.8
60 sec	42.0	45.6	44.2
2 min	35.6	42.0	40.0
5 min	26.5	32.8	31.3
30 min	9.6	9.6	8.7
60 min	8.7	8.7	6.6
3 hr	7.8	6.8	6.4
6 hr	7.8	6.8	6.4

* Heating period used to prepare specimens.

Table 33
Stress Relaxation Data at $76 \pm 1^\circ\text{F}$ and $-20 \pm 2^\circ\text{F}$
Product 9030

<u>Relaxation Period</u>	<u>Stress, psi</u>		
	<u>45 min*</u>	<u>3.0 hr</u>	<u>6.0 hr</u>
<u>Testing at $76 \pm 1^\circ\text{F}$</u>			
0 sec	3.6	3.5	3.0
10 sec	3.5	3.5	2.5
30 sec	3.5	3.0	2.5
60 sec	3.0	2.9	2.5
5 min	**	2.0	2.3
10 min	--	**	**
60 min	--	--	--
<u>Testing at $-20 \pm 2^\circ\text{F}$</u>			
0 sec	50.0	50.0	50.0
30 sec	26.5	21.4	21.9
60 sec	19.6	16.0	16.0
2 min	14.6	11.4	11.4
5 min	9.6	7.3	7.3
30 min	4.6	3.7	2.7
60 min	4.1	3.7	2.7
3 hr	3.2	2.7	2.7
6 hr	3.2	2.7	2.7

* Heating period used to prepare specimens.

** Specimens experienced adhesive failure during the test period.

Table 34
Stress Relaxation Data at $76 \pm 1^\circ\text{F}$ and $-20 \pm 2^\circ\text{F}$
Product 9005

<u>Relaxation Period</u>	<u>Stress, psi</u>		
	<u>45 min*</u>	<u>3.0 hr</u>	<u>6.0 hr</u>
<u>Testing at $76 \pm 1^\circ\text{F}$</u>			
0 sec	6.3	6.0	5.9
10 sec	3.4	3.4	2.8
30 sec	3.0	3.1	2.4
60 sec	2.9	2.9	2.3
5 min	2.4	2.4	1.9
10 min	2.3	2.3	1.6
60 min	1.8	1.8	1.3
<u>Testing at $-20 \pm 2^\circ\text{F}$</u>			
0 sec	50.0	50.0	50.0
30 sec	29.7	49.3	49.7
60 sec	48.8	48.4	48.8
2 min	47.0	45.6	45.6
5 min	42.0	39.7	41.1
30 min	28.7	28.3	26.0
60 min	27.4	22.8	21.9
3 hr	26.9	21.9	21.4
6 hr	25.6	21.4	20.5

* Heating period used to prepare specimens.

Table 35
Stress Relaxation Data at $76 \pm 1^\circ\text{F}$ and $-20 \pm 2^\circ\text{F}$
Sof-Seal

<u>Relaxation Period</u>	<u>Stress, psi</u>		
	<u>45 min*</u>	<u>3.0 hr</u>	<u>6.0 hr</u>
<u>Testing at $76 \pm 1^\circ\text{F}$</u>			
0 sec	2.8	3.0	3.0
10 sec	1.8	2.0	2.3
30 sec	1.3	1.5	2.0
60 sec	1.0	1.5	1.8
5 min	1.0	1.1	1.3
10 min	0.8	1.0	1.0
60 min	0.5	0.8	1.0
<u>Testing at $-20 \pm 2^\circ\text{F}$</u>			
0 sec	50.0	50.0	50.0
30 sec	14.1	14.8	16.6
60 sec	10.5	10.5	13.3
2 min	7.3	8.2	9.6
5 min	4.1	5.5	5.5
30 min	2.8	3.2	4.1
60 min	2.3	2.8	2.3
3 hr	2.3	2.3	2.3
6 hr	1.8	2.0	1.8

* Heating period used to prepare specimens.

Table 36
Stress Relaxation Data at $76 \pm 1^\circ\text{F}$ and $-20 \pm 2^\circ\text{F}$
Hi-Spec

<u>Relaxation Period</u>	<u>Stress, psi</u>		
	<u>45 min*</u>	<u>3.0 hr</u>	<u>6.0 hr</u>
<u>Testing at $76 \pm 1^\circ\text{F}$</u>			
0 sec	6.0	7.5	9.0
10 sec	4.0	5.0	6.0
30 sec	3.0	4.0	4.8
60 sec	2.5	3.5	4.0
5 min	2.3	1.9	2.8
10 min	1.6	1.8	2.3
60 min	1.1	1.3	1.5
<u>Testing at $-20 \pm 2^\circ\text{F}$</u>			
0 sec	50.0	50.0	50.0
30 sec	44.2	48.4	48.3
60 sec	40.2	45.4	46.5
2 min	35.1	41.9	43.8
5 min	25.6	34.4	37.4
30 min	11.0	11.7	15.1
60 min	10.0	11.1	11.9
3 hr	10.0	10.4	11.0
6 hr	9.6	10.0	11.0

* Heating period used to prepare specimens.

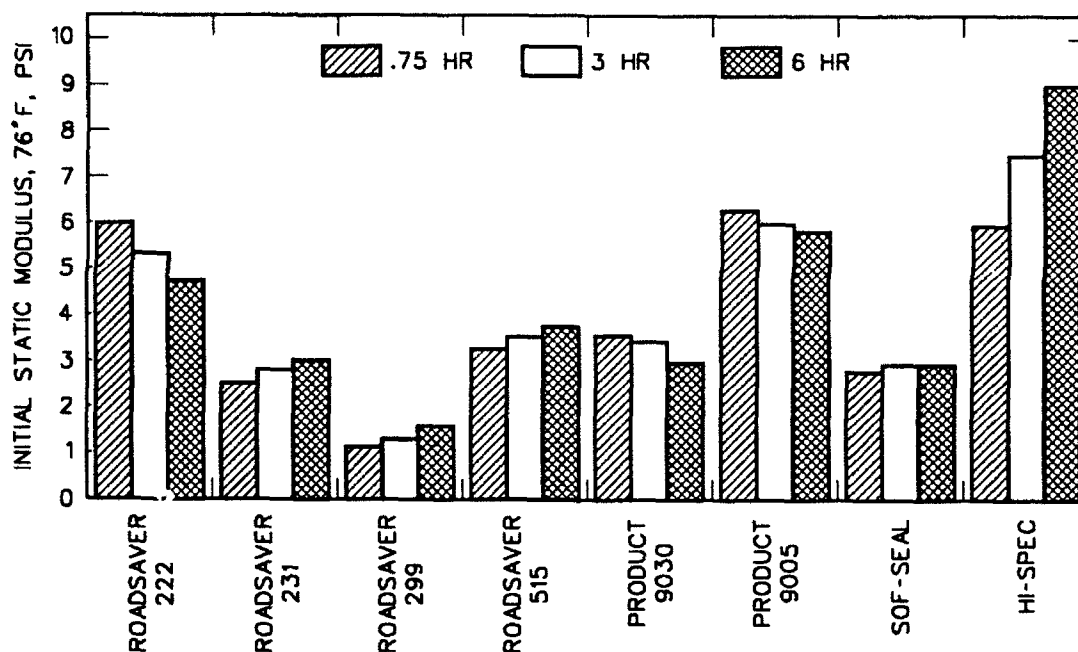


Figure 40. Initial static modulus at 76°F of non-jet-fuel-resistant sealants after 0.75, 3, and 6 hr of heating

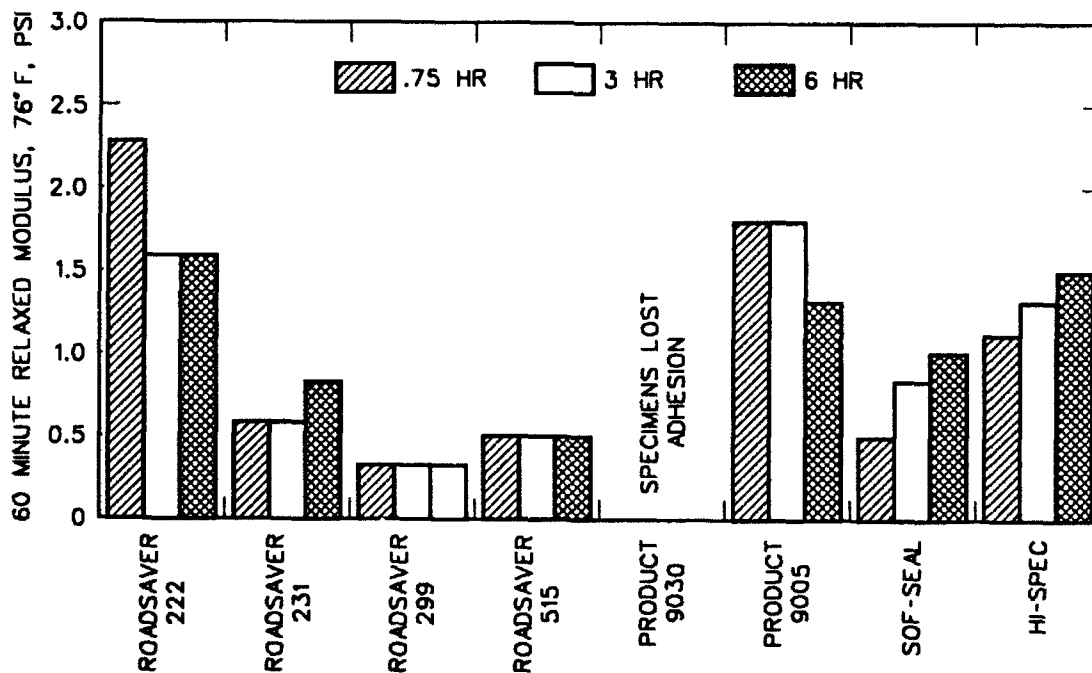


Figure 41. Static modulus at 60 min relaxation period at 76°F for non-jet-fuel-resistant sealants after 0.75, 3, and 6 hr of heating

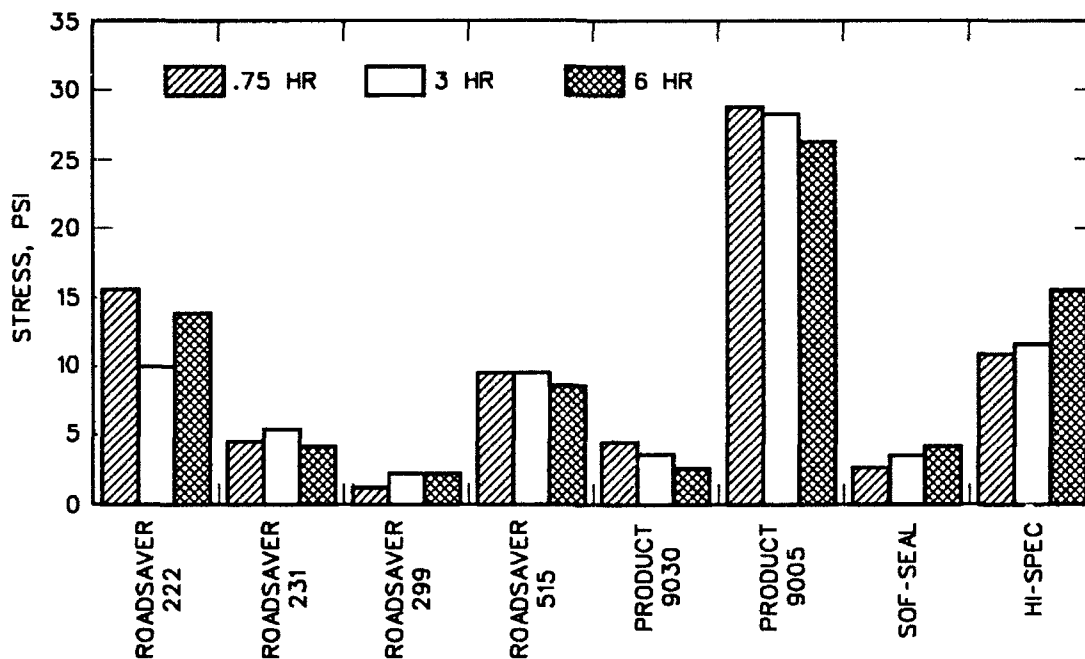


Figure 42. Stress at 30 min relaxation at -20°F for non-jet-fuel-resistant sealants after 0.75, 3, and 6 hr of heating

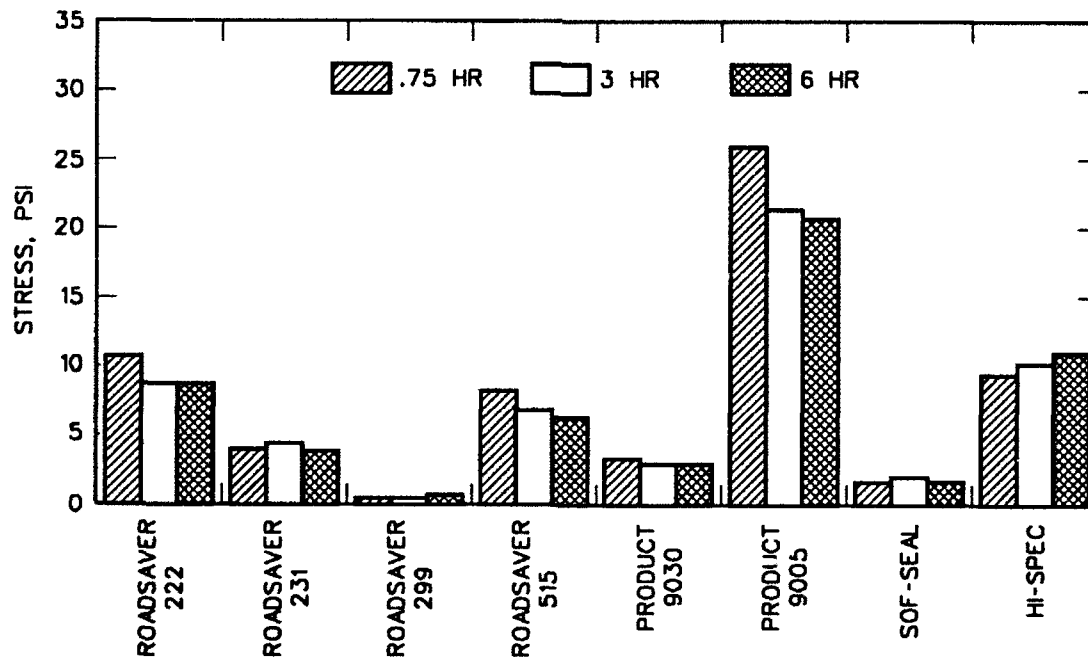


Figure 43. Stress at 6 hr relaxation at -20°F for non-jet-fuel-resistant sealants after 0.75, 3, and 6 hr of heating

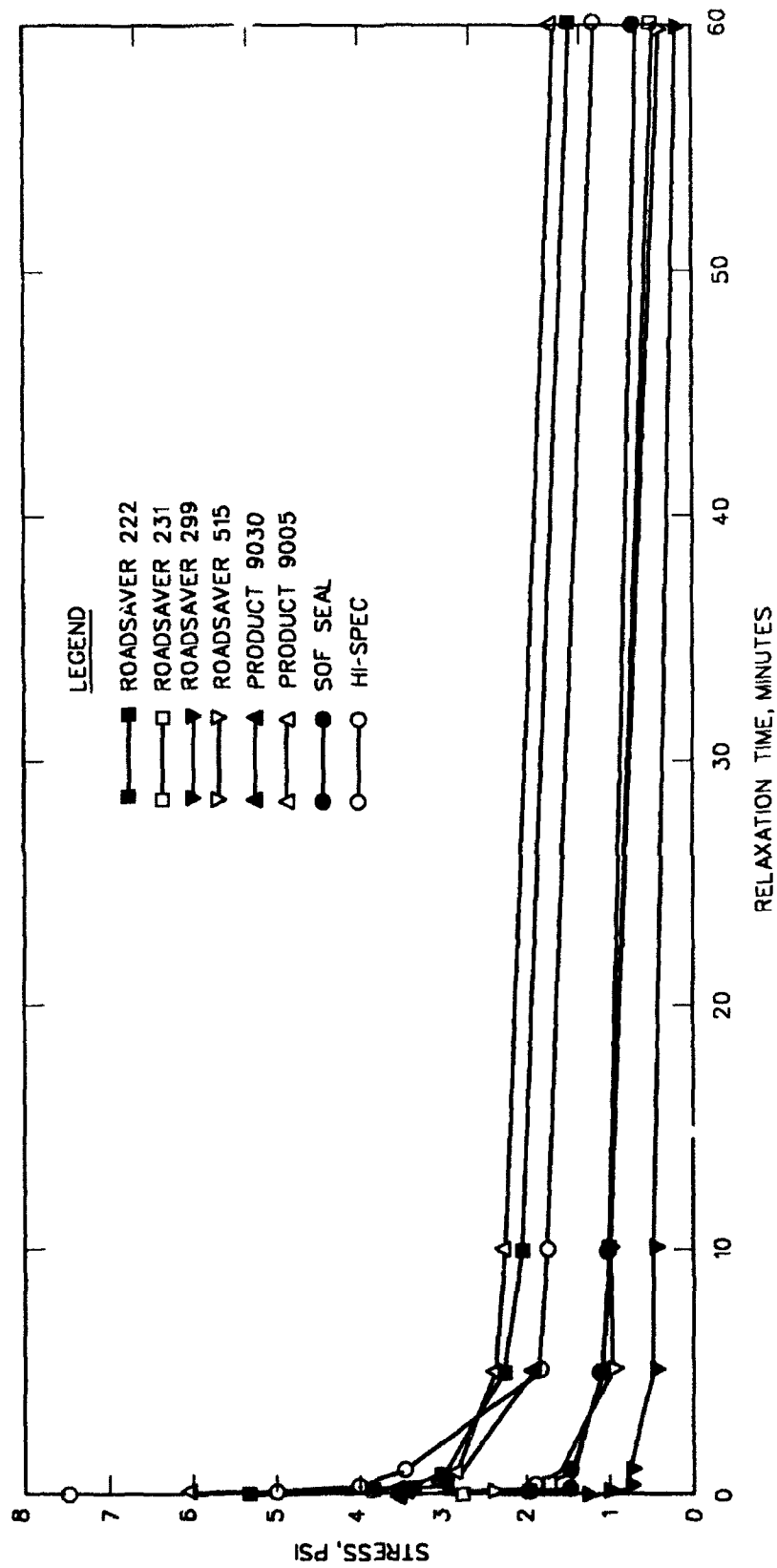


Figure 44. Stress relaxation plots at 76°F for non-jet-fuel-resistant sealants after 3 hr of heating

78. The tensile adhesion data plotted in Figure 34 varied according to the heating period and ranged from a low of 500 percent for Product 9030 after 6 hr of heating to a high of 1,100 percent for RoadSaver 231 after 3 hr of heating. RoadSaver 231 had the highest average result (1,083 percent), and RoadSaver 222 had the lowest (617 percent). Sof-Seal had the greatest change (317 percent), and RoadSaver 231 had the least (33 percent).

79. Ring and ball softening point data are plotted in Figure 35. Results varied from a low of 170°F for Sof-Seal after 45 min of heating to 216°F for RoadSaver 231 after 45 min of heating. RoadSaver 231 had the highest average result (213°F) and Sof-Seal the lowest (180°F). Product 9005, RoadSaver 299, and RoadSaver 222 were least affected by the heating period with a 3°F or less spread in results. Hi-Spec and Sof-Seal had the greatest change in results during heating (a 22°F increase for Hi-Spec and 16°F increase for Sof-Seal).

80. Ductility data at 77°F are plotted in Figure 36. Results varied from a low of 23 cm for Product 9030 after 45 min of heating to 73 cm for RoadSaver 231 after 45 min of heating. RoadSaver 231 had the highest average ductility (65 cm), and Product 9030 had the lowest (28 cm). Product 9030 had adhesive separation from the specimen mold during testing, and all other materials experienced cohesive breaks.

81. Ductility data at 39.2°F are plotted in Figure 37. Results varied from a low of 30 cm for RoadSaver 515 after 45 min of heating to a high of 91 cm for RoadSaver 231 after 45 min of heating. RoadSaver 515 had the lowest average ductility (33 cm), and RoadSaver 231 had the highest (84 cm). All specimens tested experienced cohesive breaks. RoadSaver 222, 231, Product 9030, and Product 9005 had higher ductilities at 39.2°F than at 77°F. The ductility of the other sealants were within 2 cm between temperatures.

82. Penetration data at 0°F are plotted in Figure 38. Results varied from a low of 0.07 cm for Product 9005 after 3 hr of heating to a high of 0.54 cm for RoadSaver 299 after 6 hr of heating. RoadSaver 299 had the highest average 0°F penetration (0.49 cm), and Product 9005 had the lowest (0.08 cm). The 0°F penetration for sealants which meet FS SS-S-1401C varied from 0.08 to 0.17 cm with an average of 0.13 cm. Results for the low modulus sealants varied from 0.31 to 0.49 cm with an average of 0.44 cm. The data indicate that the low-modulus sealants were softer at 0°F than sealants which meet FS SS-S-1401C.

83. Brookfield viscosity data at the sealant safe heating temperature are plotted in Figure 39. Product 9005 had the lowest average viscosity (733 cp), and RoadSaver 222 had the highest (3,563 cp). Several sealants experienced viscosity changes during heating while others were relatively constant.

84. Results of static adhesion tests revealed that all sealants except Product 9030 had satisfactory results at all heating periods. Product 9030 experienced separations in excess of 160 mm² when tested after each heating period.

85. Stress relaxation results at 76°F and -20°F are shown in Tables 29 through 36. Results are plotted in Figures 39 through 42. A plot of relaxation data at 76°F for each sealant after heating for 3 hr is shown in Figure 44. Figure 44 also illustrates the typical shape of the relaxation curves. The initial stress is the highest, and the stress decreases with time as the specimen experiences relaxation. Figure 44 also indicates that different materials not only have different initial and relaxed stresses, but that the slopes of the relaxation curves vary. The variation indicates differences in elastic characteristics. Since testing at 76°F was performed using 100 percent extension (1.00 strain), the data can also be referred to as a static modulus. Figure 40 is a plot of initial static modulus at 76°F. The data show that some sealants experience decreases in initial modulus as a result of heating while others remain approximately the same, and others experience increases. RoadSaver 299 has the lowest initial modulus (1.4 psi average), and Hi-Spec has the highest (7.5 psi average). Figure 41 shows static modulus after a 60 min relaxation period. RoadSaver 222 had the highest relaxed modulus (1.8 psi average), and RoadSaver 299 had the lowest (0.3 psi average). Product 9030 had adhesive failure during the test. Examination of the data shows that the adhesive stress at failure did not exceed 3.0 psi after 45 min of heating, 2.0 psi after 3 hr, and 2.3 psi after 6 hr of heating. Additionally, Product 9030 had the highest resilience or elasticity, experienced adhesive separations during ductility testing at 77°F, and did not pass static adhesion testing. The data indicate that sealants which meet FS SS-S-1401C have higher initial and relaxed modulus values than the other sealants tested. The 60 min relaxed modulus of RoadSaver 515 was the second lowest (0.50 psi) and was even lower than values obtained for the low modulus type materials (RoadSaver 231 and Sof-Seal). This may be due to the lower

resilience characteristics of RoadSaver 515. Sof-Seal had the largest increase in relaxed modulus with increased heating period.

86. Figure 45 is a plot of relaxation data at -20°F for the non-JFR sealants after 3 hr of heating. Due to the widely varying stiffness of the sealants, it was not possible to extend each to a uniform extension at the beginning of the test; therefore, specimens were loaded with a 50 psi tensile stress, and relaxation was monitored from that initial stress. The extension required for the different sealants to achieve 50 psi varied due to the varying stiffness of each sealant. Figure 45 shows wide variations in the relaxation plots at -20°F . Relaxed stresses after 6 hr vary from an average low of 0.6 psi for RoadSaver 299 to an average high of 22.5 psi for Product 9005. The data indicate that the majority of the relaxation occurs within approximately 1 hr after loading. Figure 42 shows stress at 30 min relaxation. The data also show that sealants which meet FS SS-S-1401C exert higher stresses than the low-modulus sealants. The effect of the heating period varies with the sealant material. Stresses after 6 hr of relaxation are plotted in Figure 43. Results follow the same trends as the 30 min data.

87. The additional test results show some major differences in the low temperature stiffness and extension characteristics of the sealants. The low-modulus sealants (RoadSaver 231, RoadSaver 299, Product 9030, and Sof-Seal) are softer and are capable of extending further at low temperatures than the sealants which meet FS SS-S-1401C. Even though the low-modulus sealants are softer, flow characteristics at 140°F and resilience results are similar to those required by FS SS-S-1401C.

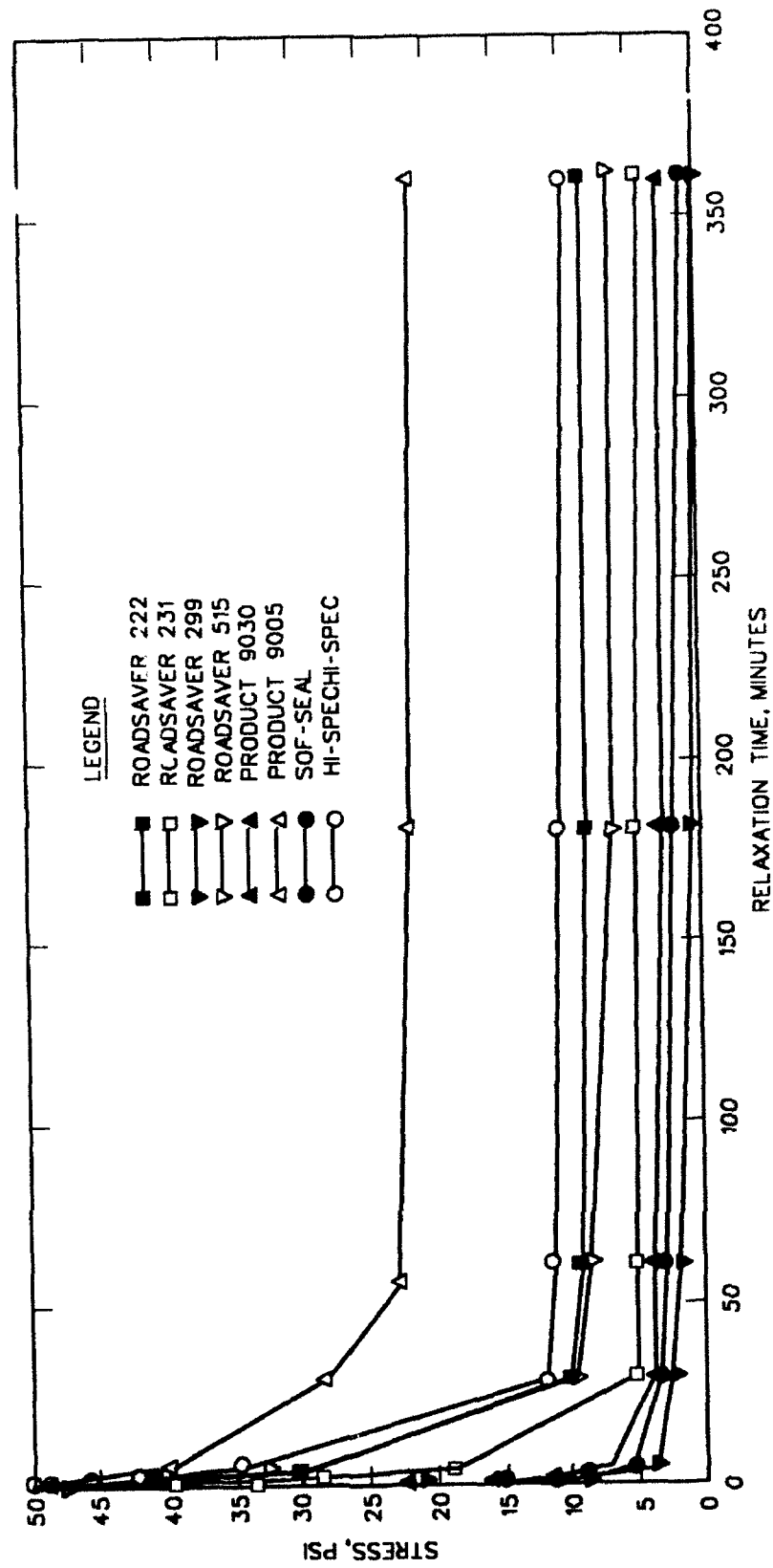


Figure 45. Stress relaxation plots at -20°F for non-jet-fuel-resistant sealants after 3 hr of heating

PART V: DEVELOPMENT OF IMPROVED HOT-APPLIED FUEL-RESISTANT SEALANT

Development Goals

88. One of the project objectives was to develop specification limits for an improved hot-applied, JFR sealant. The specific performance characteristics where improvement was desired include the following:

- a. Incompressible rejection.
- b. Weathering and durability.
- c. Low temperature properties.
- d. Blast and flow resistance
- e. Fuel resistance.

89. The testing reported in Part III of this report identified the physical properties of commercially available hot-applied, JFR sealants. The data collected during that testing provided an improved understanding of the performance limits of those sealants and is basis for the following recommended specification limits.

Incompressible rejection

90. FS SS-S-1614A does not include procedures to measure the resilient or elastic characteristics of a sealant. Inclusion of a resilience test similar to those contained in FS SS-S-1401C and FS SS-S-200E would provide that measurement. However, to include the test into a specification, the test limits must be established. The resilience data reported in Part III show that resilience values are dependent upon several factors. These factors include the sealant material, the length of the heating period, the cure period, and the aging conditions. Resilience results of the available JFR sealants varied from 50 to 72 percent after 72 hr of curing at standard laboratory conditions. Aging the samples for 7 days at 158°F caused the resilience values to decrease. Based upon these results, an initial developmental goal of 40 to 50 percent after curing at standard laboratory conditions for 72 hr would be feasible.

Weathering and durability

91. FS SS-S-1614A does not specifically address weathering and durability. The test used to evaluate these characteristics was oven-aged weight loss. Samples were taken from the sealants before and after heating. These samples were then placed in a 158°F oven and the weight loss measured

periodically. The samples taken prior to heating had weight losses of 1.9 to 2.9 percent. The samples taken after heating had weight losses ranging from 0.6 to 1.3 percent. A correlation between weight loss and durability could not be established; therefore, the testing was discontinued.

Low temperature properties

92. FS SS-S-1614A requires a sealant to withstand 50 percent extension at a test temperature of 0°F. Obvious improvements to this requirement could be to lower the test temperature, increase the amount of extension, or both. The bond testing conducted on the JFR sealants showed that none of them could be extended 50 percent extension at -20°F. Therefore, a developmental goal of 50 percent extension at -20°F was established.

Blast and flow resistance

93. FS SS-S-1614A allows a maximum flow of 3.0 cm after 5 hr exposure to 140°F. It does not include a blast resistance test. There were initially two developmental goals in this area. The first goal was to develop a material that would pass with an increase in the flow test temperature and a reduction in the allowable flow. The second goal was to determine if blast resistance could be obtained in a hot-applied JFR sealant. The laboratory testing conducted on the available JFR sealants indicated that they had significantly less flow than the 3.0 cm requirement even at higher temperatures of up to 158°F. The limit set for this developmental goal was 0.5 cm flow maximum after 24 hr exposure to 158°F.

94. Initial laboratory studies at Crafco Inc. indicated that it might be possible to formulate a hot-applied sealant that was resistant to the effects of aircraft exhaust (blast resistant). However, the sealants formulated in the laboratory to be more blast resistant were believed to be too stiff to perform satisfactorily in the field. Therefore, formulation efforts were directed toward low temperature performance improvements and away from blast resistance.

Fuel resistance

95. An improved fuel resistance would be desirable. Methods that could be used to determine improvement in fuel resistance are penetration and bond testing. The penetration tests conducted on the JFR sealants showed the amount of change between the nonimmersed penetration and the fuel-immersed penetration varied with the sealant material and the length of the heating period. Comparisons between the fuel-immersed penetration and fuel-immersed

bond showed that sealants which experienced large changes in penetration values could still pass the fuel-immersed bond testing. These results indicate that changes between nonimmersed and fuel-immersed penetrations do not necessarily indicate that adhesion is adversely affected. Therefore, a lower limit for the amount of change in penetration would not be investigated further. Instead, the fuel-immersed bond results would be used for specification consideration.

96. The surface of the oven-aged samples of JFR sealants became hardened, and the amount of hardening increased with time. This phenomena was further investigated by removing the sealant from the container and examining the properties underneath the hardened surface. This examination showed that the sealant material under the surface had not hardened, thus suggesting that the hardening was probably caused by an oxidation mechanism on the exposed surface. Subsequent to this examination, samples of several hot-applied JFR sealants which varied in age were obtained from several field installations. The samples were cut from the joints using a knife. Examination showed hardened top surfaces varying from approximately 1/8 to 1/4 in. in thickness. Hardening was also noted on the bottom surface of the sealant that was in contact with the backer rod. However, the bottom hardening was not as severe as the top. Hardening on the top of the sealant surface was large enough to cause adhesive and/or cohesive separations to the depth of the hardened material. Below the hardened surface, strong adhesion to the joint surfaces was observed. In areas where the sealant thickness was approximately 1/4 in., the total thickness of the sealant had hardened, and both adhesive and cohesive failure modes were observed. The hardened surfaces observed in field specimens and the laboratory oven-aged specimens had a similar hardened, brittle appearance. These observations indicate that surface hardening may be a primary cause of failure in hot-applied, JFR sealants. It was decided that a method to measure surface hardening characteristics should be developed.

97. Penetration samples of Superseal 1614A, Plant 1, Product 9012, and NEA 1614 were prepared and subjected to forced-draft oven exposure at 158°F for 4 weeks. Penetration at 77°F was monitored during the 4 week period to determine surface hardening characteristics. The results are shown in Table 37 and plotted in Figure 46. These data show that forced-draft oven exposure of the specimens cause significant drops in penetration. The

Table 37

Cone Penetration of Jet-Fuel-Resistant Sealants Subjected
to Forced-Draft Oven Aging at 158°F

<u>Exposure Period</u>	<u>Sealant</u>					
	<u>Product 9012</u>		<u>NEA 1614</u>		<u>Plant 1</u>	
	<u>Data*</u>	<u>Percent Retained</u>	<u>Data</u>	<u>Percent Retained</u>	<u>Data</u>	<u>Percent Retained</u>
24 hr cure **	0.93	100	1.07	100	1.27	100
1 day oven aged	0.66	71	0.76	71	0.75	59
3 day	0.31	33	0.40	37	0.46	36
7 day	0.24	26	0.26	24	0.32	25
14 day	0.19	20	0.19	18	0.19	15
28 day	0.19	20	0.20	19	0.20	16

* All results are reported in centimetres.

** All specimens were prepared using the standard FS SS-S-1614A 3 hr heating period.

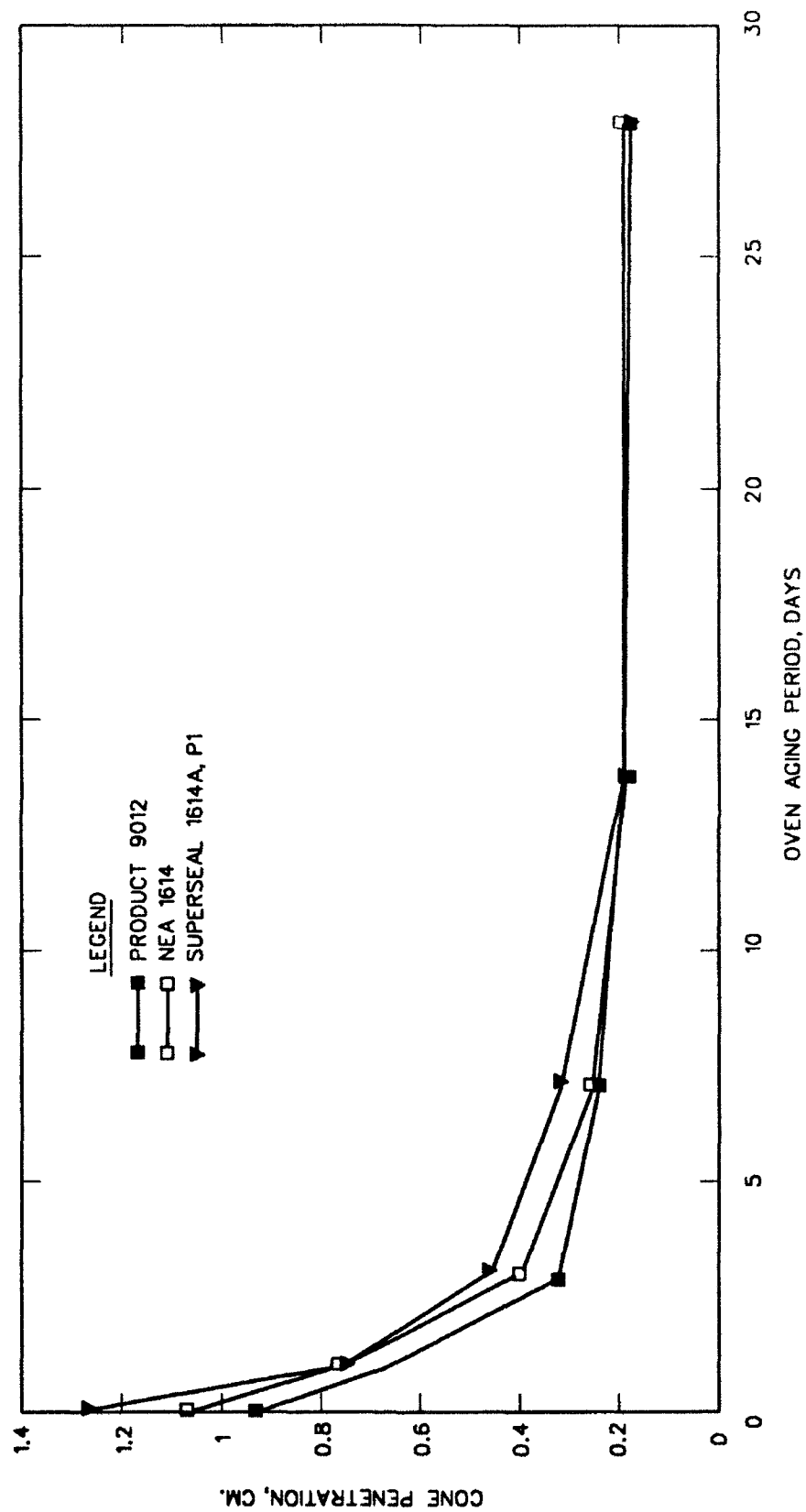


Figure 46. Penetration of jet-fuel-resistant sealants when subjected to forced-draft oven aging at 158°F

majority of the drop occurs within 3 days of exposure. Each of the three sealants tested experienced similar decreases in penetration. Average percent retained penetration after 1 day was 67 percent and 35 percent after 3 days. Further exposure for a total of 14 days showed an average result of 18 percent. Subsequent to this evaluation, samples of Nitoseal 777 were tested in the same manner. Results showed retained penetrations of 89 percent after 1 day, 68 percent after 3 days, and 42 percent after 7 days. Based on these results, it was decided that a development goal for improved sealant durability would be to provide materials which would be more resistant to aging (i.e. had a greater percent retained penetration when exposed to oven aging at 158°F for 7 days).

Laboratory Research

98. Laboratory experimentation was performed in an attempt to meet the development goals. During the development phase for the JFR phase of the project, approximately 200 laboratory mixtures were prepared and tested. A wide variety of different types and percentages of sealant raw materials were studied. Formulation efforts were directed to produce sealant materials which could meet the development goals, thus verifying that the goals were achievable. A realistic specification for improved materials would then be developed. The laboratory research effort also included formulation studies directed to provide performance levels beyond the development goals to determine if more stringent requirements could be considered.

99. During the laboratory research, several key sealant materials which had some of the desired improved characteristics were produced. A summary of the characteristics for key mixtures that were developed through the laboratory research are shown in Table 38. The exact formulations are not provided due to provisions in the research agreement between WES and Crafco. Information in Table 38 shows that all of the key mixes, except CP-23, were capable of passing bond testing at -20°F. Aged penetration retention results for mixtures CP-23 and CP-34 were 40 and 41 percent, respectively. These results are slightly improved compared to the three FS SS-S-1614A materials which had

Table 38

Physical Properties of Key Fuel-Resistant Sealant Materials

<u>Property</u>	<u>CP-23</u>	<u>CP-34</u>	<u>CP-63</u>	<u>CP-73</u>	<u>CP-74</u>	<u>CP-87</u>	<u>CP-93</u>
Penetration, cm							
Nonimmersed	1.45	1.46	1.43	1.58	1.40	1.63	1.58
Fuel-immersed	1.38	1.22	1.25	1.16	1.06	1.18	1.29
Aged penetration							
Retention (72 hr), percent	40	41	84	85	73	80	81
Flow, 158°F, cm	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Bond, -20°F, 50 percent							
Nonimmersed	Fail 3	Pass 3	Pass 3	Pass 3	Pass 3	Pass 3	Pass 3
Fuel-immersed	Pass 3	Pass 3	Pass 3	Pass 3	Pass 3	Pass 3	Pass 3
Resilience, 77°F, percent	65	76	52	76	74	52	41

This is a significant improvement compared to the original FS SS-S-1614A mixtures which had a 35 percent average.

100. Oven-aged penetration data for Superseal 1614A, Plant 1 and mixture CP-73 are shown in Table 39. Testing was performed at 158°F using specimens cured at laboratory standard conditions for 5 days prior to aging. Data are plotted in Figure 47. Results show that Superseal 1614A, Plant 1 experienced a large decrease in penetration after 3 days of oven aging (36 percent retained). Mixture CP-73, however, retained 105 percent of its original penetration after 3 days of aging. Mixture CP-73 experienced a large drop in penetration between 14 and 30 days of aging from 93 to 21 percent retained. These results indicate that mixture CP-73 experienced much less hardening than Superseal 1614A, Plant 1 when subjected to 14 days of forced-draft oven aging at 158°F. After 30 days aging, results show that mixture CP-73 had hardened significantly, but it had a 50 percent higher penetration than Superseal 1614A, Plant 1.

101. Based on examination of mixtures and results, it was decided that mixture CP-87 would be used as the basis for specification development and for production mixtures for the field installation. "As supplied" properties of mixture CP-87 are shown in Table 40. These results show that mixture CP-87 is a liquid material with typical "as supplied" viscosity when compared to data for existing liquid JFR sealants in Table 6. "As supplied" oven-aged weight loss results are also similar to results obtained for existing sealants. Physical properties as measured by FS SS-S-1614A of mixture CP-87 after 45 min, 3, 4.5, and 6 hr of heating are provided in Table 41. These results indicate conformance to all requirements of FS SS-S-1614A except for the non-immersed penetration after each heating period. Data show relatively uniform results during heating as indicated by penetration which changed by 0.11 cm (7.0 percent) during heating.

102. Table 42 shows the additional physical properties of mixture CP-87. Discussions of each property follow.

- a. Resilience after 24 hr of curing was uniform during the 6 hr heating period and varied from 48 to 50 percent with an average of 49 percent.

Table 39
158°F Over-Aged Penetration for Superseal 1614A,
Plant 1 and Experimental Mixture CP-73

<u>Aging Period</u>	<u>Superseal 1614A, Plant 1</u>		<u>CP-73</u>	
	<u>Penetration</u>	<u>Retained Percent</u>	<u>Penetration</u>	<u>Retained Percent</u>
Original	1.05	100	1.28	100
1 day	0.62	59	1.51	117
3 day	0.38	36	1.34	105
7 day	0.31	30	1.26	98
14 day	0.23	22	1.19	93
30 day	0.15	14	0.27	21
60 day	0.11	10	0.16	13

Note: All penetration data are in centimetres. Data were obtained in 3-oz tins, and specimens were conditioned at standard laboratory conditions for 5 days before initiating aging exposure.

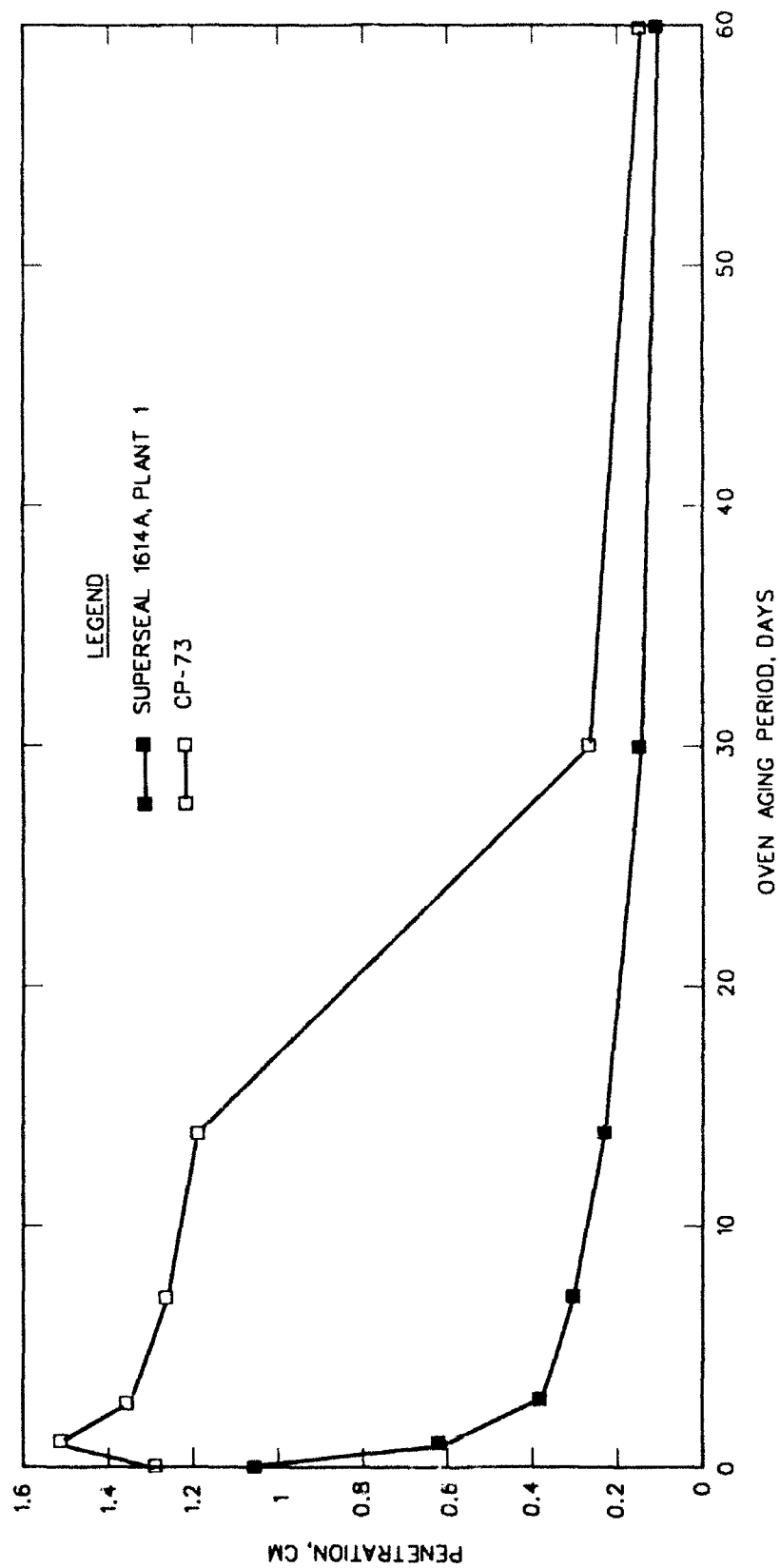


Figure 47. Oven-aged penetration at 158°F for Superseal 1614A, Plant 1 and mixture CP-73

Table 40
"As-Supplied" Properties of Mixture CP-87

<u>Parameter</u>	<u>Result</u>
Brookfield viscosity, cps	
77°F (No. 4 at 20 rpm)	9,600 cps
100°F (No. 2 at 20 rpm)	2,200 cps
Oven aged weight changes, 6 oz containers, 158°F, percent	
2 hr	-0.1
6 hr	-0.2
24 hr	-0.6
72 hr	-0.9
168 hr	-2.0
Oven aged weight change, 5 1/2 in. (144 mm) pan, 158°F, percent	
2 hr	-0.5
6 hr	-1.0
24 hr	-2.4
72 hr	-3.6
168 hr	-6.8

Table 41

Test Results for Mixture CP-87 in Accordance with FS SS-S-1614AWhen Heated for 45 min, 3, 4.5, and 6 hr

<u>FS SS-S-1614A Test</u>	<u>45 min</u>	<u>3 hr</u>	<u>4.5 hr</u>	<u>6 hr</u>
Penetration, cm				
Nonimmersed	1.55	1.49	1.46	1.44
Fuel-immersed	1.20	1.14	1.09	1.08
Change	-0.35	-0.35	-0.37	-0.36
Fuel immersed				
Weight change, percent	0.11	0.11	0.15	0.19
Flow, cm	0.0	0.0	0.0	0.0
Bond				
Nonimmersed	S*	S	S	S
Fuel-immersed	S	S	S	S
Water immersed	S	S	S	S

* S signifies satisfactory performance in accordance with specification requirements.

Table 42
Additional Physical Characteristics of Mixture CP-87

Test	Heating Period			
	45 min	3.0 hr	4.5 hr	6.0 hr
Resilience, 24 hr, percent	48	50	49	49
72 hr, percent	60	59	61	60
Aged 72 hr Resilience				
24 hr, 158°F, percent	49	51	57	53
168 hr, 158°F, percent	53	55	55	55
Penetration, 0°F, cm				
Nonimmersed	18	13	11	13
Fuel-immersed	35	33	32	30
Flow, cm				
158°F, 5 hr	0.0	0.0	0.0	0.0
158°F, 24 hr	0.0	0.0	0.0	0.0
200°F, 5 hr	0.0	0.0	0.0	0.0
200°F, 24 hr	0.0	0.0	0.0	0.0
Oven-aged weight loss, percent				
24 hr, 158°F	-0.3	-0.3	-0.3	-0.2
72 hr, 158°F	-0.4	-0.4	-0.4	-0.4
168 hr, 158°F	-0.6	-0.5	-0.5	-0.5
Nonimmersed bond				
0°F, 100 percent, 3 cycles	S*	S	S	S
-20°F, 50 percent, 3 cycles	S	S	S	U**
-20°F, 100 percent, 3 cycles	U	U	U	U
Flame resistance	U	U	U	U
Tack free time, min	18	15	15	12
Viscosity, Brookfield, 270°F, cps	7,300	7,600	8,400	8,800

* S signifies satisfactory performance in accordance with the test specification.

** U signifies unsatisfactory performance.

- b. Resilience after 72 hr of curing was also relatively uniform during heating varying from 59 to 61 percent. The average result was 60 percent.
- c. The 24 hr aging at 158°F produced decreases in resilience. The resilience varied from 49 to 57 percent with an average of 53 percent for all heating periods. Drops in resilience ranged from 4 to 11 percent compared to the unaged samples.
- d. The 168 hr aging produced smaller changes in resilience than for the previously tested sealants. The change varied from a decrease of 2 percent to an increase of 4 percent. Average data for all heating periods were 54 percent which is an average increase of 1 percent compared to the 24 hr aged results.
- e. Penetration at 0°F ranged from 0.18 to 0.11 cm and decreased during heating. Results are in the same general range as for the existing tested JFR sealants.
- f. Fuel-immersed penetration at 0°F ranged from 0.35 to 0.30 cm and decreased during heating. These results are higher than those obtained for the existing tested JFR sealants.
- g. Flow testing at 158 and 200°F for both 5 and 24 hr produced 0.0 cm of flow which is similar to most of the tested existing sealants.
- h. Oven-aged weight loss data after exposure to 158°F for 168 hr show losses which range from 0.6 percent to 0.5 percent. This loss was less than for the existing sealants tested.
- i. Nonimmersed bond testing showed satisfactory results for all heating periods at 0°F with 100 percent extension. Testing at -20°F with 50 percent extension showed satisfactory results for each heating period except 6 hr. Unsatisfactory results were obtained at -20°F with 100 percent extension for all heating periods. These results indicate improved low temperature performance compared to the existing sealants.
- j. Flame resistance testing showed unsatisfactory results at each heating period. Results are similar to the tested existing sealants.
- k. Tack free time varied from 12 to 18 min. Results are in the same range as the tested existing sealants.
- l. Viscosity at the safe heating temperature varied from 7,300 to 8,800 cp during heating. Average viscosity was 8,025 cp. These results are in the same range as the existing tested sealants. Mixture CP-87, however, only had a 20.5 percent viscosity increase during heating. In comparison, the existing tested sealants showed increases from 48.5 to 83.3 percent. This indicates that mixture CP-87 is more stable during heating than the existing tested sealants.

103. Stress relaxation data at 76°F and -20°F are tabulated in Table 7. Data at 76°F show an initial static modulus which varies from 3.2 to 4.1 psi with an average of 3.8 psi. The modulus increased 29 percent during heating

from 45 min to 6 hr. Results show that mixture CP-87 had a lower average initial static modulus at 76°F, and increased less during heating than the existing JFR sealants. The 60 min relaxed modulus varied from 1.5 psi to 1.7 psi with an average of 1.6 psi. The increase during heating was 13.3 percent.

104. Thirty minute relaxation results at -20°F (Table 43) range from 2.3 to 7.3 psi and average 4.9 psi. This indicates that mixture CP-87 exerts significantly lower stresses than the existing tested sealants. The stresses of the existing sealants ranged from 8.2 to 20.9 psi. Relaxation data after 6 hr show an average result of 3.2 psi which is also significantly lower than the existing tested sealants which had average results ranging from 6.6 to 15.2 psi.

Suggested Specification

105. Considering the laboratory results for the experimental mixtures, a specification for an improved hot-applied, single component, JFR sealant was developed. The specification is patterned after the current FS SS-S-1614A specification with several changes in property limits and test procedures. The specification is listed in Appendix B.

106. A comparison of methods and requirements of FS SS-S-1614A and the suggested JFR specification is shown in Table 44. The suggested specification requires increased high temperature flow resistance, increased hardening resistance, increased low temperature extension characteristics, and increased elasticity or resilience. The differences between the suggested specification and FS SS-S-1614A are as follows:

- a. Specimen curing. All specimens are cured at standard laboratory conditions for 72 hr prior to initiating testing procedures.
- b. Nonimmersed penetration. Upper limit is 1.60 cm instead of 1.30 cm maximum. This permits use of a softer material.
- c. Fuel-immersed penetration. Upper limit is 1.60 cm maximum and no increase is permitted over the nonimmersed result. No softening is permitted after fuel immersion.
- d. Aged penetration retention. This is a new requirement with a 70 percent of the initial value minimum. This requirement measures increased resistance to hardening during aging.

Table 43
Stress Relaxation Data at $76 \pm 1^\circ\text{F}$ and $-20^\circ\text{F} \pm 2^\circ\text{F}$.
Mixture CP-87

<u>Relaxation Period</u>	<u>Stress, psi</u>		
	<u>45 min*</u>	<u>3.0 hr</u>	<u>6.0 hr</u>
<u>Testing at $76 \pm 1^\circ\text{F}$</u>			
0	3.2	4.1	4.0
10 sec	2.3	3.0	3.0
30 sec	2.0	2.8	2.7
60 sec	1.9	2.7	2.5
5 min	1.7	2.2	2.2
10 min	1.6	2.1	2.1
60 min	1.5	1.7	1.7
<u>Testing at $-20 \pm 2^\circ\text{F}$</u>			
0	50.0	50.0	50.0
30 sec	45.6	47.7	47.5
60 sec	42.0	45.0	45.2
2 min	33.3	40.9	39.7
5 min	20.5	29.5	30.6
30 min	2.3	5.0	7.3
60 min	1.8	3.6	5.9
3 hr	1.8	3.6	4.1
6 hr	1.8	3.6	4.1

* Signifies heating period used to prepare specimens.

Table 44

Comparison of Requirements of FS SS-S-1614A and the Suggested Specification
for Improved Hot-Applied JFR Sealant

<u>Test</u>	<u>FS SS-S-1614A Requirement</u>	<u>Suggested Improved Requirement</u>
Heating period	3 hr at safe heating temperature	Same
Specimen curing	Standard conditions, time not specified	72 \pm 4 hr at standard conditions*
Nonimmersed penetration	1.30 cm max	1.60 cm max
Fuel-immersed penetration	1.55 cm max, 0.25 cm max increase	1.60 cm max, shall not exceed nonimmersed
Aged penetration retention	No requirement	70 percent min
Change in mass by fuel-immersion	2.0 percent max	1.0 percent max
Flow	140°F, 5 hr, 3.0 cm max flow	158°F, 24 hr, 0.3 cm max flow
Bond to concrete	All tests at 0°F, 50 percent extension, 3 cycles	All tests at -20°F, 50 percent extension, 3 cycles
Resilience	No requirement	45 percent min

* 73° \pm 4°F with 50 \pm 5 percent relative humidity.

- e. Change in mass by fuel immersion. The limit is 1.0 percent maximum instead of 2.0 percent maximum.
- f. Flow. Testing is performed at 158°F for 24 hr and the limit is 0.3 cm maximum.
- g. Bond to concrete. Testing is at -20°F instead of 0°F to measure low temperature extension and flexibility characteristics.
- h. Resilience. This is a new requirement which would be conducted using the same procedures described in ASTM D3406. The percent recovery shall be a minimum of 45 percent. This measures the sealants ability to reject incompressibles. FS SS-S-1614A has no requirement.

107. Since test procedures in the suggested specification are similar to current FS SS-S-1614A procedures, laboratory testing according to the suggested specification would require only slight modifications. Laboratories equipped to perform FS SS-S-1614A and FS SS-S-1401C analyses could perform the required tests in accordance with the proposed specification with no additional equipment.

Production Results

108. A pilot production batch of sealant based on mixture CP-87 was manufactured for installation in the field test project. The sealant was tested in accordance with requirements of the suggested specification as an improved hot-applied JFR sealant. Results and specification limits are shown in Table 45. Results indicate that the sealant from the pilot production batch met the suggested specification requirements.

Table 45

Suggested JFR Specification Test Results of
Mixture CP-87 Pilot Production Batch

<u>Test</u>	<u>Results</u>	<u>Specification Requirements</u>
Nonimmersed penetration	13.5	16.0 mm max
Fuel-immersed penetration	11.3	Not greater than nonimmersed
Aged penetration retention	87 percent	70 percent min
Change in mass by fuel-immersion	-0.1 percent	± 1.0 percent max
Flow	0.0	3.0 mm max
Bond to concrete		
Nonimmersed	Satisfactory	Satisfactory
Fuel-immersed	Satisfactory	Satisfactory
Water immersed	Satisfactory	Satisfactory
Resilience	65 percent	45 percent min

PART VI: DEVELOPMENT OF IMPROVED HOT-APPLIED
NON-JET-FUEL-RESISTANT SEALANT

Development Goals

109. The purpose of this phase of the project was to develop specification limits for a hot-applied, non-JFR sealant with improved low temperature performance characteristics compared to current materials which meet FS SS-S-1401C. There are several sealant materials available which are similar in characteristics to materials meeting FS SS-S-1401C, except that they have higher penetrations and improved low temperature extension characteristics. Specifications for these materials are generally referred to as "low-modulus ASTM D3405." Several states have issued these types of specifications including Iowa (Iowa Department of Transportation 1984), South Dakota (South Dakota Department of Transportation, undated), Kansas (Kansas Department of Transportation 1986), Michigan (Michigan Department of Transportation 1987), and Minnesota (Minnesota Department of Transportation 1988). The major development goal of this phase was to provide a material specification which would require improved low temperature sealant performance. The requirements would be similar to the performance level of low-modulus D3405 materials while maintaining adequate high temperature performance.

110. Tests performed and reported in Part IV of this report were used to identify and compare physical properties of current FS SS-S-1401C materials and several of the low-modulus materials. Review and comparison results contained in Part IV indicate the following:

- a. Bond testing. Results showed the low-modulus materials were capable of passing bond testing at -20°F using 200 percent extension while the FS SS-S-1401C materials did not. Results were similar for both nonimmersed and water immersed testing.
- b. Tensile adhesion. Results show that the low-modulus materials have higher tensile adhesion than FS SS-S-1401C materials. Product 9030, however, which meets several of the low-modulus specifications, had lower tensile adhesion results compared to the other low-modulus materials.
- c. Flow. Flow testing results at 140°F using the FS SS-S-1401C procedures show the low-modulus materials met current requirements.
- d. Resilience. Results show that the low-modulus materials can conform to current FS SS-S-1401C requirements of 60 percent minimum. Other types of data obtained indicate that increased

resilience can adversely affect other properties. Examination of stress relaxation data shows that the lower resilience materials relax to lower stresses than the higher resilience materials. Additionally, high resilience may also adversely affect adhesion characteristics.

- e. Penetration. Results show that the low-modulus materials have higher penetrations than the FS SS-S-1401C materials.

Suggested Specification

111. Based on the results contained in Part IV of this report and the desired development goals, a suggested specification for low-modulus, hot-applied, non-JFR sealant was developed. The specification is contained in Appendix C. A summary of requirements and differences between FS SS-S-1401C and the suggested specification is contained in Table 46. Differences are as follows:

- a. Penetration. The suggested limit is 9.0 to 15.0 mm compared to 9.0 mm maximum for FS SS-S-1401C. This requirement permits the use of a softer material.
- b. Flow. The requirement is the same at 3.0 mm maximum.
- c. Resilience. The requirement is 50 to 80 percent compared to 60 percent minimum for FS SS-S-1401C. The lower limit of 50 percent permits materials with increased ability to undergo relaxation, and the upper limit of 80 percent limits material elasticity.
- d. Indentation. The requirement is 1.0 to 3.5 mm compared to 0.5 to 1.5 mm. The higher range allows the use of a softer material.
- e. Bond to concrete. The suggested procedure is to extend the samples to 200 percent elongation instead of the 50 percent requirement of FS SS-S-1401C. This procedure measures increased low temperature extension and flexibility characteristics.
- f. Static adhesion. The suggested specification includes a static adhesion requirement to further evaluate sealant adhesion characteristics.
- g. Compatibility. Compatibility results are the same as for FS SS-S-1401C.

Table 46

Comparison of Requirements of FS SS-S-1401C and the Suggested
Specification for Low-Modulus Hot-Applied
Non-JFR Sealant

<u>Test</u>	<u>FS SS-S-1401C Requirement</u>	<u>Suggested Low-Modulus Requirement</u>
Heating period	3 hr at safe heating temperature	3 hr at safe heating temperature
Penetration	9.0 mm max	9.0-15.0 mm
Flow	3.0 mm max	3.0 mm max
Resilience	60 percent min	50-80 percent
Indentation	0.5-1.5 mm	1.0-3.5 mm
Aged resilience	60 percent min	50-80 percent
Indentation	0.5-1.5 mm	1.0-3.5 mm
Bond to concrete	50 percent at -20°F 3 cycles	200 percent at -20°F 3 cycles
Static adhesion	None	Pass
Compatibility	Pass	Pass

Production Results

112. After several laboratory mixtures were made and tested, a pilot production batch of sealant to meet the suggested low-modulus specification was made. The mixture code for this mixture was 230-3.

113. Test results for mixture 230-3 in accordance with FS SS-S-1401C after 45 min, 3 hr, and 6 hr of heating are tabulated in Table 47. Data shows that mixture 230-3 meets all requirements of FS SS-S-1401C except for penetration and initial indentations.

114. Additional test results on mixture 230-3 are provided in Table 48. Results show that mixture 230-3 passed bond testing at -20°F using 200 percent extension, and static adhesion had an average tensile adhesion of 994 percent. Penetration at 0°F averaged 36 which is softer than for materials tested in accordance with FS SS-S-1401C. Ductility at 77°F averaged 70 cm, and at 39.2°F, results averaged 88 cm. Viscosity was reduced from 950 cp to 650 cp during heating.

Table 47
FS SS-S-1401C Test Results for Mixture 230-3
after Heating for 45 min, 3, and 6 hr

<u>FS SS-S-1401C Test</u>	<u>45 min</u>	<u>3 hr</u>	<u>6 hr</u>
Penetration, mm	14.7	12.7	12.5
Flow, mm	0.0	0.0	0.0
Resilience, percent	66	73	71
Indentation, mm	3.0	2.8	2.0
Aged resilience, percent	66	67	68
Indentation, mm	2.6	2.6	2.6
Bond			
Nonimmersed	S*	S	S
Water immersed	S	S	S
Compatibility	S	S	S

* S signifies satisfactory performance in bond and compatibility testing in accordance with FS SS-S-1401C requirements.

115. Stress relaxation results at 76°F and -20°F are provided in Table 49. Results show an average initial static modulus at 76°F of 2.4 psi which is lower than results for the FS SS-S-1401C materials. The 60 min relaxed modulus at 76°F averages 0.9 psi which is also lower than for the FS SS-S-1401C materials. Results at -20°F show an average 30 min stress of 4.2 psi, and a 6-hr relaxed stress of 2.7 psi, which is lower than for the FS SS-S-1401C materials.

116. Table 50 shows conformance of the pilot production batch of mixture 230-3 with the suggested low-modulus specification.

Table 48
Additional Physical Characteristics of Mixture 230-3

Test	Heating Period		
	45 min	3 hr	6 hr
Bond, nonimmersed			
-20°F, 100 percent, 3 cycles	S*	S	S
-20°F, 200 percent, 3 cycles	S	S	S
Bond, water immersed			
-20°F, 100 percent, 3 cycles	S	S	S
-20°F, 200 percent, 3 cycles	S	S	S
Tensile adhesion, percent	1,000	975	1,008
Softening point, °F	200	209	209
Ductility, 77°F,			
5 cm/min, cm	70	71	69
Ductility, 39.2°F,			
5 cm/min, cm	87	88	88
Penetration, 0°F, cm	0.37	0.34	0.38
Viscosity, Brookfield,			
410°F, cps	950	850	650
Static adhesion	Pass	Pass	Pass

* S signifies satisfactory performance in the bond test using FS SS-S-1401C evaluation criteria.

Table 49
Stress Relaxation Data at $76 \pm 1^\circ\text{F}$ and $-20^\circ\text{F} \pm 2^\circ\text{F}$
Mixture 230-3

<u>Relaxation Period</u>	<u>Stress, psi</u>		
	<u>45 min*</u>	<u>3.0 hr*</u>	<u>6.0 hr*</u>
<u>Testing at $76 \pm 1^\circ\text{F}$</u>			
0	1.7	2.9	2.6
10 sec	1.5	1.5	1.9
30 sec	1.4	1.3	1.7
60 sec	1.3	1.2	1.5
5 min	1.1	1.0	1.3
10 min	1.0	0.9	1.2
60 min	0.9	0.8	1.0
<u>Testing at $-20 \pm 2^\circ\text{F}$</u>			
0	50.0	50.0	50.0
30 sec	35.6	41.1	42.0
60 sec	27.4	36.5	37.4
2 min	20.5	28.3	29.7
5 min	9.1	16.8	9.2
30 min	3.2	5.5	4.1
60 min	3.2	3.7	3.2
3 hr	3.2	2.7	3.2
6 hr	2.7	2.7	2.7

* Signifies heating period used to prepare specimens.

Table 50

Results for Pilot Production Batch of Mixture 230-3 When Tested in
Accordance with the Suggested Specification for Low-Modulus
Sealant, Joint, Non-Jet-Fuel-Resistant, Hot-Applied for
Portland Cement and Asphalt Concrete Pavements

<u>Requirement</u>	<u>Result</u>	<u>Specification Limits</u>
Penetration, mm	12.7	9.0 - 15.0
Flow, mm	0.0	3.0 max
Resilience, percent	73	50 - 80
Indentation, mm	2.8	1.0 - 3.5
Aged resilience, percent	67	50 - 80
Indentation, mm	2.6	1.0 - 3.5
Bond to concrete,		
Nonimmersed	Satisfactory	Satisfactory
Water immersed	Satisfactory	Satisfactory
Static adhesion	Satisfactory	Satisfactory
Compatibility	Satisfactory	Satisfactory

PART VII: DEVELOPMENT OF ENCAPSULATING PRIMER

Literature Search

117. Two literature searches were conducted to gather information on potential primer systems which could be used as encapsulants. The first search was restricted to patents at the US Patent Office. The search was directed to identify any patents in which primer or surface coatings were applied to concrete to reduce moisture passage and to improve adhesion of sealing materials. This search was unsuccessful as no patents meeting the search criteria were found.

118. The second search effort was conducted by the Arizona State University (ASU) First Division of Hayden Library at ASU and the Information Center, Information Technology Laboratory at WES. The computer search used several sources including (a) Dialog Information Service, (b) Patent files from 1950 to 1991, (c) Trade Industry Index, (d) New Product Announcements, and (e) World Coating and Surface Abstracts. The search identified a variety of US and foreign patents related to surface sealing of concrete, and other construction materials and references related to adhesion and coatings were identified. However, no references that referred to encapsulating primer treatments prior to joint sealing were located.

Analysis of the CRD-C 525-89 Bubble Testing Procedure

119. The US Army Corps of Engineers developed a test method and specification for evaluating bubbling tendencies of hot-applied sealants. This procedure was issued in December, 1989 as CRD-C 525-89, "Standard Specification for Bubbling Tendencies of Hot-Applied Joint Sealers for Rigid Pavements" (US Army Corps of Engineers Handbook of Concrete and Cement 1989). The procedure consists of exposing samples which have passed nonimmersed bond testing to 24 hr at 158°F in a forced-draft oven and then evaluating for evidence of bubbling. Previous testing has shown that some hot-applied sealants experience bubbling when tested using this procedure.

120. In order to gain a better understanding of the bubble testing procedure, initial testing with a variety of sealants, substrates, and a coating was performed. Sealants used include Crafcro RoadSaver 211 (an ASTM D1190

materials, RoadSeal 222 (S-1401C material), Crafcro 34515 (Modified AASHTO M173), and Superseal 1614A, Plant 1. Substrates used were 1/4 by 2 by 3 in. steel plates, 1/4 by 2 by 3 in. glass plates, and standard concrete bond test blocks which were either oven dried to a constant weight or dried to a saturated surface dried (SSD) condition.

121. Testing with steel and glass plates was performed using the CRD-C 525-89 procedure except that steel or glass plates were used instead of concrete blocks. These specimens were not subjected to bond testing prior to initiating the 158°F oven exposure period. Test results showed that none of the sealants bubbled or swelled when steel or glass plates were used.

122. Sets of oven dried concrete blocks were prepared by drying to constant weight in a $250 \pm 10^\circ\text{F}$ forced-draft oven. Average weight loss data for the blocks were 5.1 percent after 3 hr, 5.6 percent after 5 hr, 6.0 percent after 21 hr, and 6.0 percent after 24 hr. Data show that the standard FS SS-S-1401C concrete blocks contain approximately 6 percent moisture when they are in a saturated surface dry condition. After the 24 hr drying period, the blocks were stored and cooled in a desiccator until use. Specimens for each of the four sealants were poured, and were then subjected to 24 hr of exposure at 158°F. Again, as with the metal and glass plates, no evidence of bubbling or swelling was observed.

123. Testing as specified in CRD-C 525-89 was then performed with concrete blocks prepared in accordance with FS SS-S-1401C and FS SS-S-1614A. These blocks were prepared as saturated surface dried and then subjected to required blotting prior to pouring specimens. The blocks experienced a weight loss which averaged 0.5 percent due to the required blotting and drying which left an average of 5.5 percent moisture in the blocks when sealant was poured to make the test specimens. A deviation from the CRD-C 525-89 procedure was that bond testing was not performed prior to aging the specimens at 158°F for 24 hr. Results showed significant amounts of expansion, flow, and swelling of each sealant. More sealant flow and deformation were noted for the non-JFR sealants than for the JFR Superseal 1614A, Plant 1 which only experienced swelling.

124. Examination of data for the steel, glass, oven-dried, and saturated surface dry block substrates shows that bubbling occurred only with blocks which contained moisture. It appears that bubbling is caused by the release of moisture vapor from the concrete which then migrates through the

sealant at high temperatures. At high temperatures such as 158°F, hot-applied sealants are softened and may not be capable of resisting moisture vapor migration.

125. Additional tests were performed in which the moisture content of concrete blocks was adjusted to 3.0 percent, 1.0 percent, and 0.5 percent by oven drying, resoaking and then redrying to achieve the proper moisture content. Testing was performed with RoadSaver 515. Results showed significant amounts of expansion, flow, and swelling of the sealant at each of the three moisture contents. These results show that concrete blocks with a moisture content as low as 0.5 percent can cause swelling and flow when testing is performed using CRD-C 525-89 procedures.

126. Several of the additional sealants received from other manufacturers were also tested for bubbling tendencies using the basic CRD-C 525-89 procedure as described above. The additional sealants tested were Products 9030 and 9005, Sof-Seal, and Hi-Spec. Each of these sealants experienced swelling, flow, and deformation during the 24 hr oven exposure period.

127. During testing it was noted that the amount of sealant deformation, swelling, and flow that occurred using CRD-C 525-89 did not appear to be typical of bubbling which occurs in field installations. Therefore, the test temperature was reduced from 158°F to 140°F. Additional tests were conducted using a test temperature of 120°F. The bubbling experienced in the laboratory using the test temperature of 120°F was representative of bubbling exhibited in the field. Sealant bubbling which occurs in field installations typically does not produce flow of the sealant, but shows up as numerous individual bubbles on top of the sealant surface. These bubbles may range from approximately 1/16 to 1/4 in. in diameter. Test temperatures of 120 and 140°F were used with Superseal 1614A, Plant 1 and 140°F with RoadSaver 515. Results showed that Superseal 1614A, Plant 1 did not experience any bubbling, swelling, or flow at 120 or 140°F. RoadSaver 515 experienced formation of several small bubbles on the top of the sealant surface and experienced swelling.

128. While testing was proceeding with various sealants, temperatures, etc., several block coatings were tried to provide some basic information on encapsulating coatings. Prepared concrete blocks were coated on the bonding surface side with a two-component epoxy glue thickened by adding approximately 1 percent amorphous fumed silica. Following curing, bond specimens were prepared and tested for bubbling. Sealants used were the same as those used in

the initial bubble testing. Results for tests with these coated blocks showed similar amounts of expansion, flow, and deformation as with the uncoated blocks. Comparisons were made by estimating the height and width of the swelled volume. Additional tests were performed with a two-component epoxy floor paint as a block coating and using RoadSaver 515. Swelling, deformation, and flow occurred when this combination was tested.

129. An initial trial at making an encapsulating primer was made. This primer, coded RS-1, had a solid content of 27 percent and a viscosity of 140 cp at 75°F. Concrete blocks with moisture contents of 0.5, 1.0, and 3.0 percent were brush coated using RS-1 and tested for bubbling tendencies using RoadSaver 515. Results after 24 hr of oven aging at 158°F showed similar expansion amounts as with the uncoated blocks at 3.0 and 1.0 percent moisture. However, no swelling was observed for the primed blocks at the 0.5 moisture content. This indicates that primer coatings offer promise for reducing bubbling. During testing, it was noted that samples with primed blocks which had moisture contents of 3.0 and 1.0 percent appeared to swell at a lesser rate when exposed up to approximately 6 hr at 158°F. This also indicates that the primer may reduce bubbling.

130. Additional tests with RS-1 were performed by applying two coats of the primer with 1 hr of drying between coats. This was done to provide a thicker surface coating to better encapsulate the blocks surfaces. Testing was performed using RoadSaver 515 and saturated surface dried blocks following the method as previously described. Results showed swelling, flow, and deformation after 24 hr of exposure; however, the amount of swelling was less than for unprimed blocks and occurred initially at a slower rate. These results indicate that the thickness of the applied surface coating may influence the amount of bubbling which occurs.

Bubbling Analysis Procedure

131. As a result of the initial testing using CRD-C 525-89, it became apparent that a more quantitative method of studying sealant bubbling and expansion would be of assistance compared to visual examinations and measurement of height and width of swell. A procedure was developed to monitor the volume of sealant swelling during the 24 hr specimen exposure to forced-draft oven aging. Basically, the procedure consists of monitoring sealant expansion

by making an impression casting of the swelled sealant surface using a quick setting dental impression compound, and then measuring the swell volume by filling the impression with water. In this manner, the volume of swell can be determined and a quantitative measure of primer or coating effectiveness can be made.

132. The swell volume measurement test procedure consisted of the following steps:

- a. Prepare bond test samples in accordance with FS SS-S-1401C or SS-S-1614A. If primed blocks were used, prime and cure the blocks prior to preparing the bond test samples.
- b. Place the bond test samples in the oven at 158°F (or other temperature which is being studied) in accordance with CRD-C 525-89.
- c. After the exposure period, remove the specimen from the oven. Place a square mask which measures 1/2 in. high by 2 1/2 in. by 3 in. inside dimensions on top of the specimen centered over the sealant (Figure 48). Then immediately fill the mask with a quick setting dental impression compound (such as Jelt-rate Alginate Impression Material, Type II - Regular Set, supplied by L. D. Caulk Division, Dentsply International, Inc., Milford, Delaware 19963-0359) and strike off level with the top surface. Allow the compound to set (approximately 1 min).
- d. Carefully remove the impression from the mask, place the impression on a scale with the sealant impression facing up and immediately weigh to the nearest 0.1 g.
- e. Fill the void in the impression created by the sealant with water and determine the weight of water required to fill the void.
- f. The weight of water required to fill the void can also be expressed as the void volume, or swelled sealant volume in cubic centimeters.
- g. If further exposure to oven temperatures is desired, place the specimen in the oven immediately after removal of the mask and impression.

133. An example of swelling data measured using this procedure is shown in Figure 49 which is a plot of the swell volume for Roadsaver 515 with saturated surface dried blocks tested at 140 and 158°F. Swell volume was monitored after 2, 6, and 24 hr of exposure. Data show that after 24 hr of exposure at 158°F, Roadsaver 515 swelled 6.0 cc, and after 24 hr exposure at 140°F, it swelled 2.9 cc.

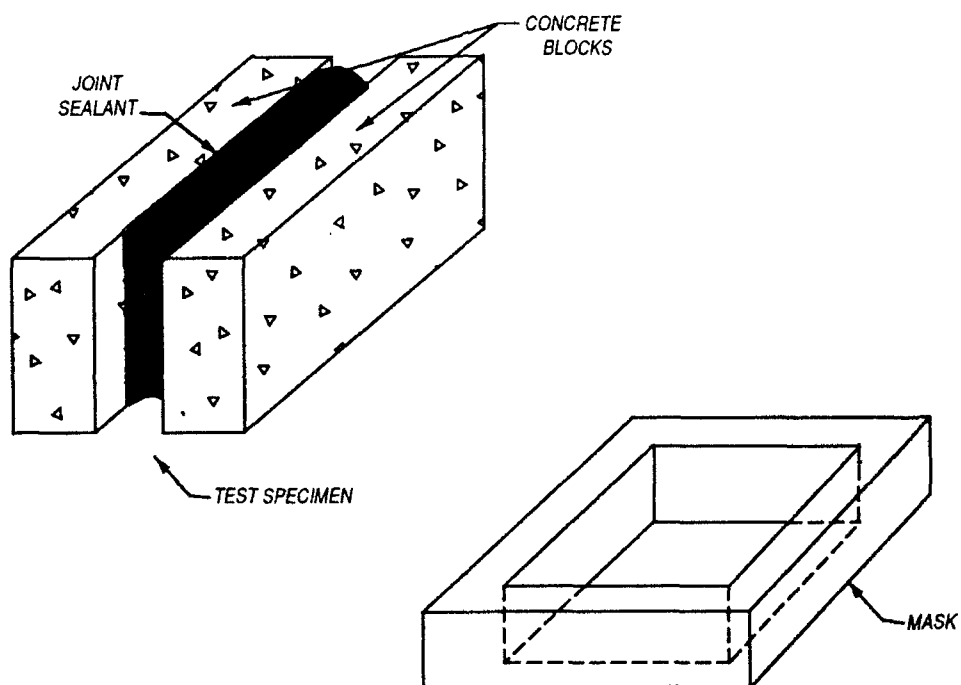


Figure 48. Swell volume determination

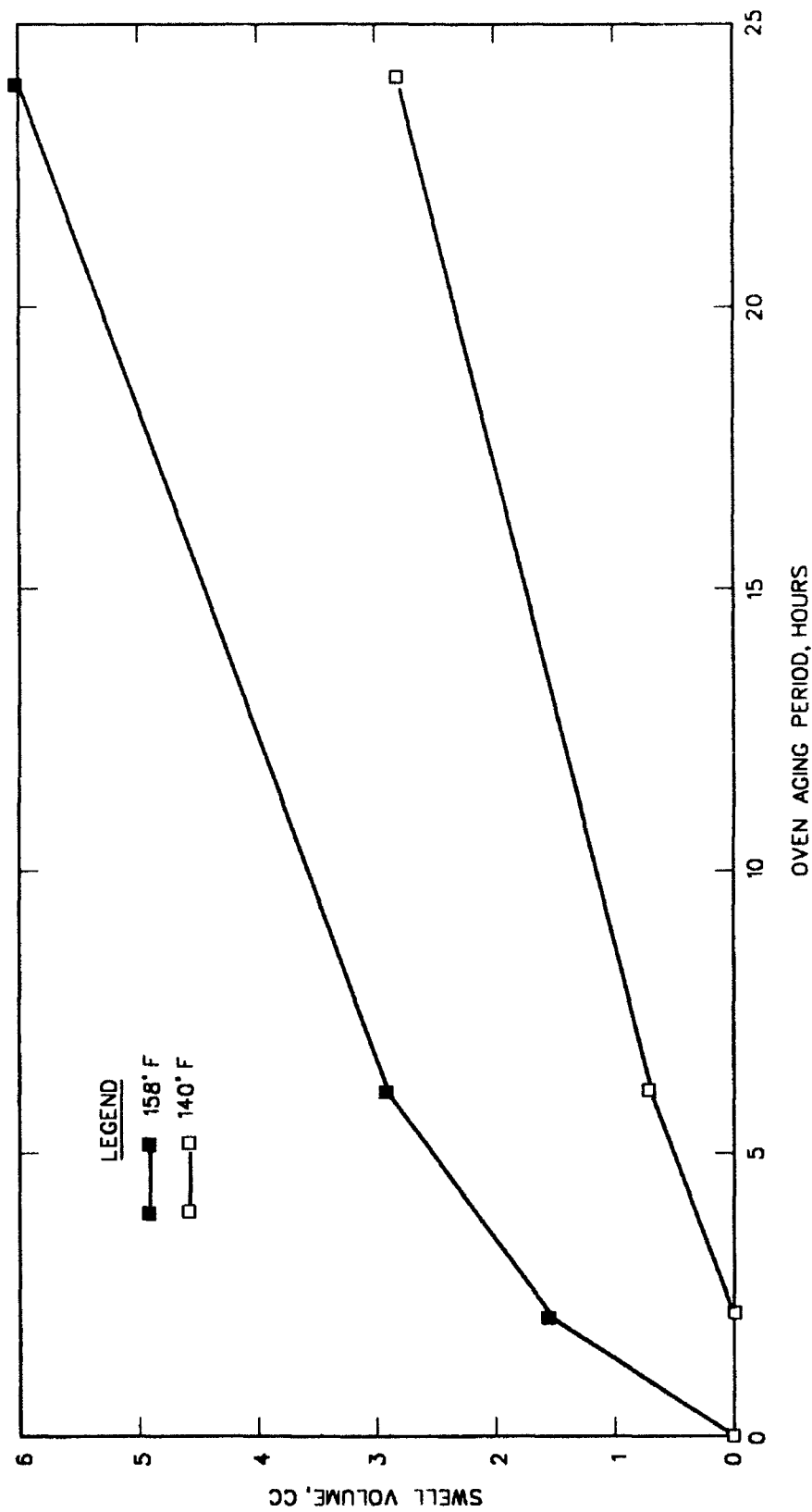


Figure 49. Swell volume at 140 and 158°F for RoadSaver 515 using unprimed saturated surface dried concrete blocks

Jet-Fuel-Resistant Primer Formulation Studies

134. Laboratory research was conducted to develop encapsulating primer materials for use with JFR sealants. Early in the project, it was decided that solvent based materials would probably not be appropriate for use with JFR sealants due to solubility of the primer residues. Therefore, efforts were directed at investigating two-component systems. Development goals for the two-component primer material for use with JFR sealants are to:

- a. Provide a surface coating to encapsulate the concrete to reduce moisture related bubbling.
- b. Provide a surface coating which would not detrimentally affect adhesion, and ideally would improve adhesion while maintaining required fuel-resistance.
- c. Provide material which is simple and easy to apply and cures shortly after application (preferably less than 1 hr).

135. During the laboratory research phase, 40 different mixtures of raw materials were studied as potential primer systems. The mixtures included a variety of polymeric systems with different curing systems and resinous additives. Several two-component, solvent-based systems were investigated. Many of the materials did not cure in the desired time period to be considered acceptable. Also, the two-component systems studied did not provide an acceptable sealant adhesion, especially after fuel-immersion.

136. Data in Table 51 are a summary of swelling and adhesion data for several of the JFR primers tested. Data are also included for one coat and three coats of Nitoprime 23 which is used with Nitoseal 777 for adhesion improvements. This is a commercially available primer supplied by Fosroc International, LTD. Swell data plotted in Figure 50 show that several of the primer materials reduced swelling of the sealant, and that PF-22 eliminated swelling. One coat of Nitoprime 23 did not influence swelling; however, three coats reduced swelling by approximately one-half after 24 hr exposure. The experimental primer formulations reduced sealant adhesion properties in nonimmersed and fuel-immersed bond testing, and reduced tensile adhesion results. Blocks coated with Nitoprime 23 produced tensile adhesion results similar to the unprimed blocks. Nonimmersed bond results were satisfactory; however fuel-immersed bond results for blocks treated with Nitoprime 23 were unsatisfactory due to failed areas greater than the permitted 160 mm².

Table 51
Results for Jet-Fuel-Resistant Primers with
Superseal 1614A, Plant 1

Test	Unprimed	PF-22	PF-30	PF-33	Nitoprime	
					1 Coat	3 Coats
158°F swell volume, cc*						
2 hr	0.3	0.0	0.3	0.7	0.2	0.3
6 hr	0.4	0.0	0.7	0.7	0.3	0.3
24 hr	0.9	0.0	1.0	1.1	0.8	0.5
Bond testing**						
Nonimmersed	S†	U††	U	U	S	S
Fuel-immersed	S	U	U	U	U	U
Tensile adhesion, percent‡	750C	300A	400A	400A	750C	800C

* Swell data are obtained using the procedure in paragraphs 109 and 110 of this report.

** Testing is performed using FS SS-S-1614A procedures.

† S signifies satisfactory bond test results.

†† U signifies unsatisfactory results in accordance with FS SS-S-1614A requirements.

‡ Test performed using ASTM D3583 procedures. A signifies adhesive failure; C signifies cohesive failure.

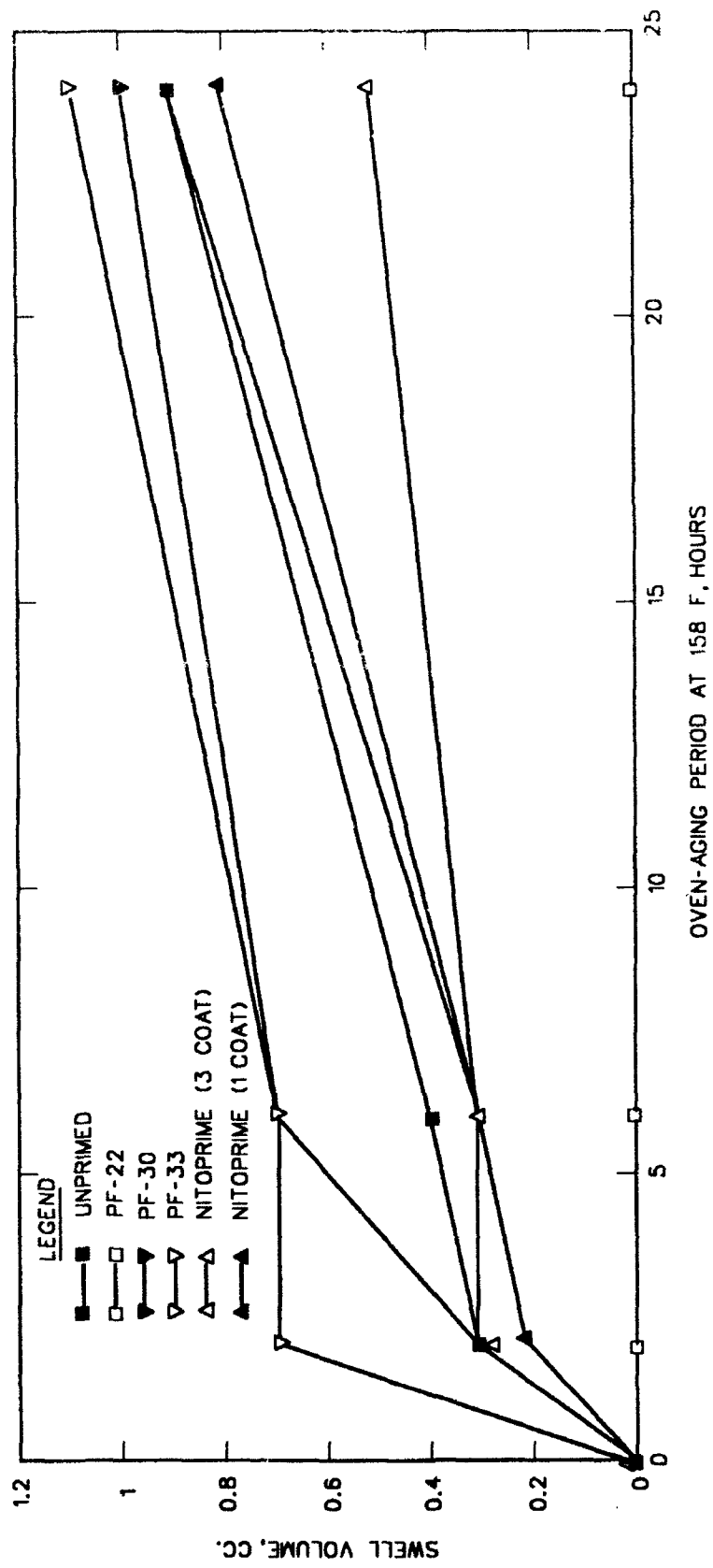


Figure 50. Swell volume at 158°F for Superseal 1614A, Plant 1 using unprimed and primed saturated surface dried concrete blocks

137. The research work performed was not successful in developing acceptable encapsulating primer materials for use with JFR sealants.

Non-Jet-Fuel-Resistant Primer Formulation Studies

138. Laboratory testing was conducted to develop encapsulating primer materials for use with the non-JFR sealants. During initial project planning meetings, it was decided that development efforts would be directed at investigating single-component systems. Development goals for the single-component non-JFR primer were to:

- a. Provide a surface coating to encapsulate the concrete to reduce moisture related bubbling.
- b. Provide a surface coating which would not detrimentally affect adhesion.
- c. Provide a material which is simple and easy to apply and cures shortly after application (preferably less than 1 hr).

139. During the laboratory research phase, approximately 20 different mixtures of raw materials were studied as potential primer systems. The mixtures included combinations of polymeric materials, resinous additives, and a variety of additives in a solvent carrier. The materials were formulated of 25 to 35 percent solids and typically cured well within 1 hr after application to concrete blocks.

140. Table 52 contains a summary of swelling and adhesion data for several of the non-JFR primers. Swelling data obtained at 158°F are plotted in Figure 51. Figure 51 shows that the primer coatings reduced swell volume of RoadSaver 515 throughout the 24 hr aging period. Reductions in swelling after 24 hr average 34 percent and ranged from 22 to 50 percent for the primers reported in Table 52. After 6 hr of aging at 158°F, reductions in swell volume averaged 60 percent and ranged from 38 to 69 percent. Data show that primer RS-12 improved adhesion of RoadSaver 515 as indicated by an approximate 30 percent increase in tensile adhesion test results and changed the failure mode from adhesive to cohesive.

141. The research work performed produced primer coating materials which partially met the development goals for non-JFR primer. Swelling in laboratory testing was reduced, adhesion to concrete was increased, and the produced primer materials were low viscosity materials that could be easily applied and cured quickly.

Table 52
Results for Several Non-Jet-Fuel-Resistant Primers
With RoadSaver 515

Test	Unprimed	RS-2	RS-3	RS-6	RS-7	RS-12	3 Coats
							RS-12
158°F swell volume, cc							
2 hr	1.5	1.1	1.4	0.0	0.0	0.3	0.3
6 hr	2.9	1.4	1.8	0.9	1.0	1.2	0.6
24 hr	6.0	3.6	4.7	4.5	3.0	4.3	3.7
Bond testing*							
Nonimmersed	S**	--	--	--	--	S	S
Water immersed	S	--	--	--	--	S	S
Tensile adhesion, percent†	850A	--	--	--	--	1100C	--

* Testing is performed using FS SS-S-1401C procedures.

** S signifies satisfactory bond test results in accordance with FS SS-S-1401C requirements.

† Test performed using ASTM D3583 procedures. A signifies adhesive failure; C signifies cohesive failure.

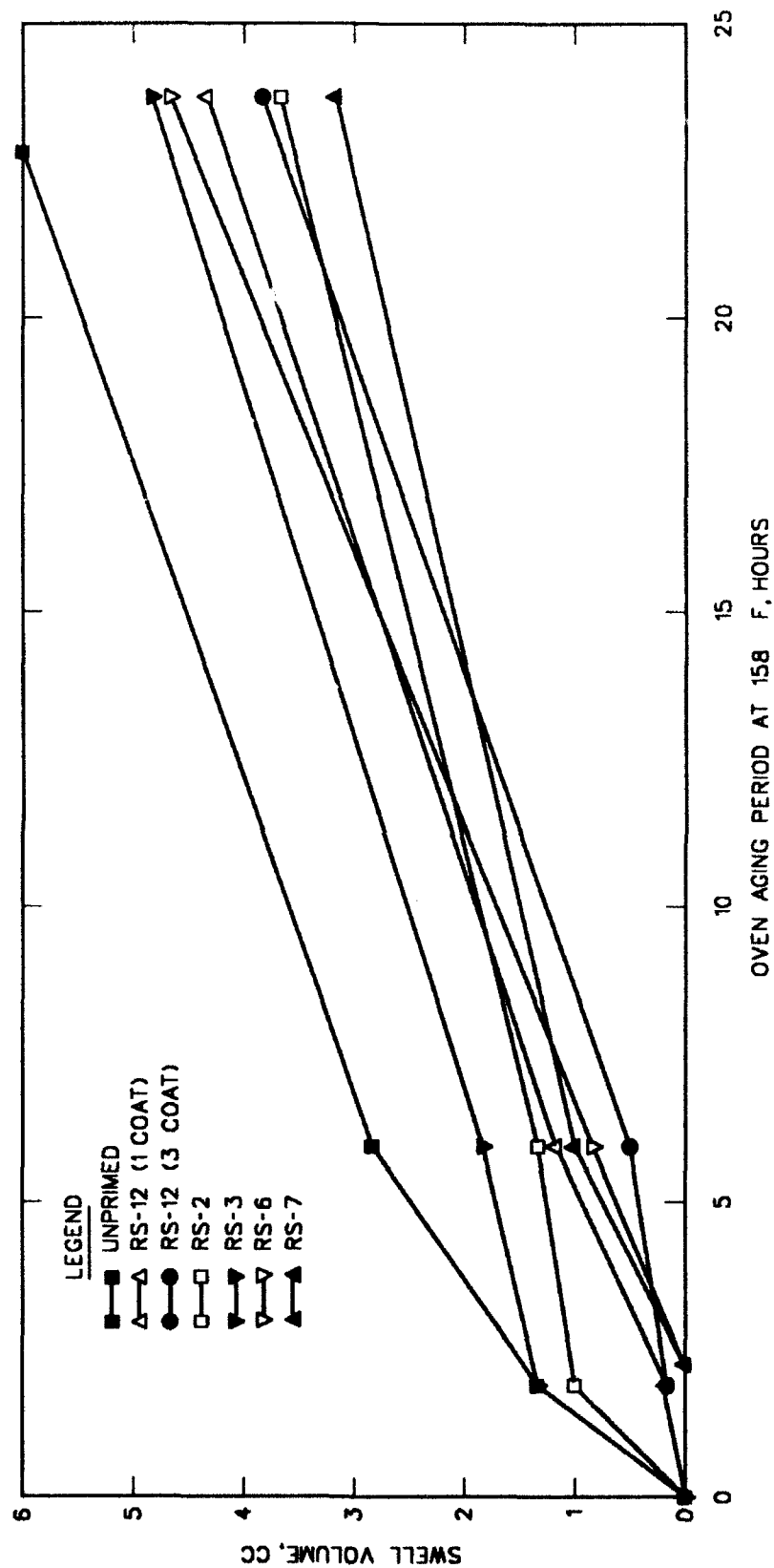


Figure 51. Swell volume at 158°F for RoadSaver 515 using unprimed and primed saturated surface dried concrete blocks

Suggested Specification for Non-Jet-Fuel-Resistant Primer

142. Based on the laboratory results for non-JFR primers, a suggested specification for primer, joint, non-JFR, cold-applied, single-component for portland cement concrete pavements was developed. The specification is contained in Appendix D. Requirements of the specification are based on properties and characteristics of primer mixtures reported herein. A summary of requirements of the suggested specification is as follows:

- a. Viscosity. Viscosity of the primer is measured at 77°F and indicates the consistency of the material for application purposes. The suggested requirement is 25 to 250 cp.
- b. Tack-free time. Tack-free time is measured using a modification of the basic FS SS-S-200E procedure with primer that has been applied to a prepared standard concrete block. The suggested requirement is 10 min maximum. The requirement indicates a material which cures quickly after application to concrete.
- c. Cured tack-free evaluation. The tack-free characteristics are measured on primer coated concrete block specimens which have been allowed to cure for 60 min. The procedure then subjects the cured primer to a tack-free evaluation using a 60 min period at 158°F. The suggested requirement is that at the end of the 60 min period, the weight is removed, and the polyethylene strip shall pull free without any primer adhering to the strip. This evaluation indicates the high temperature stability of the primer coating.
- d. Nonvolatile content. The nonvolatile content indicates the residue content of the primer. The suggested requirement is 20 to 40 percent.
- e. Permeability. The permeability requirement indicates the ability of the primer coating to limit passing of moisture. The procedure subjects oven-dried, primer coated concrete blocks to water immersion and measures the amount of water absorption into the block expressed as a percentage of the saturated surface dried moisture content of the block. The suggested limit is 10 percent maximum. Testing performed during development of the procedure showed that unprimed concrete blocks experience water absorption which exceeds 95 percent when tested using the specified procedure.
- f. Low temperature flexibility. The procedure uses the ASTM C711-72 "Low Temperature Flexibility and Tenacity of One-Part, Elastomeric, Solvent-Release Type Sealants" procedure with several modifications. The requirement indicates flexibility of the cured primer residue at 30°F.
- g. Adhesion. Adhesion of the primer is evaluated using primer coated concrete blocks for sealant bond testing with manufacturer recommended sealants. The suggested requirement is that

the sealant shall pass bond test requirements with the primer coated blocks.

Production Results

143. A pilot production batch of primer to meet the suggested specification was made for test section installations with non-JFR sealants. The batch was based on mixture RS-12. Properties of the production primer batch when tested in accordance with the suggested specification are shown in Table 53. Results indicate that the pilot production batch of primer met the requirements of the suggested specification.

Table 53

Primer Mixture RS-12 Pilot Production Batch Test Results

<u>Test</u>	<u>Result</u>	<u>Specification Limits</u>
Viscosity	68 cp	25-250 cp
Tack-free time	4 min	10 min max
Cured tack free	Pass	Pass
Nonvolatile content	25.6 percent	20-40 percent
Permeability	2.9 percent	10 percent max
Low temperature flexibility	Pass	Pass
Adhesion	Pass	Pass

PART VIII: CONCLUSIONS AND RECOMMENDATIONS

144. Evaluations of the procured hot-applied JFR sealants indicated that these sealants met FS SS-S-1614A requirements and were capable of passing low temperature bond testing at 0°F, but were not capable of passing at -20°F. Results also showed that these materials had significant surface hardening when subjected to oven aging at 158°F. Flow testing revealed that all materials had lower degrees of flow than the maximum requirement in FS SS-S-1614A. Testing performed at various heating periods showed that physical properties of hot-applied JFR sealants changed with changes in the heating period. This indicates that if the materials are over heated or exposed to prolonged heating in the field, premature failure due to hardening can occur.

145. Evaluations of the procured hot-applied non-JFR sealants showed that low-modulus ASTM D3405 materials were capable of passing 200 percent extension bond testing at -20°F while materials manufactured to meet FS SS-S-1401C were not. The flow and resilience properties of the low-modulus materials were similar to FS SS-S-1401C materials. Additional testing procedures showed the sealants had differences in adhesion and modulus characteristics. Test results show that different heating periods cause the properties of some materials to change more than others. This indicates that the low-modulus type materials could perform more satisfactorily in cold climates than the FS SS-S-1401C materials. This possibility will be evaluated during the field evaluations.

146. Laboratory development work revealed that it was possible to produce hot-applied JFR sealant materials which could satisfactorily pass non-immersed, water immersed, and fuel-immersed bond testing at -20°F, with less surface hardening when subjected to oven-aging at 158°F than current materials. A suggested specification for this type of sealant with improved performance characteristics was developed in accordance with the first objective of the project. The developed material should have better low temperature properties than current hot-applied materials and should be more resistant to aging. These properties could translate to a longer lasting seal. The field evaluations will be used to determine exact life-cycle benefits.

147. A suggested specification for low-modulus non-JFR sealant was developed for sealants with improved low temperature properties in accordance with the second objective of the project. The specification includes bond

testing at -20°F using 200 percent extension, a static adhesion evaluation, and resilience limits in addition to requirements of FS SS-S-1401C.

148. Testing using the CRD-C 525-89 bubbling tendencies procedures revealed that hot-applied sealants do not experience bubbling when in contact with metal, glass, or oven-dried concrete surfaces. Bubbling, however, occurs when these sealants are in contact with concrete surfaces which contain as low as 0.5 percent moisture by weight of the concrete. This indicates that moisture is one of the major causes of bubbling and that bubbling caused by moisture may be difficult to eliminate. The difficulty arises from the fact that in most areas of the United States, the natural moisture content of the concrete may be greater than 0.5 percent.

149. Appropriate adhesion with hot-applied JFR sealants could not be established with the developmental fuel-resistant primer materials studied. However, primer materials for use with non-JFR sealants were developed which reduced sealant bubbling in the laboratory and provided increased sealant adhesion to portland cement concrete. A suggested specification for the primer materials was developed in accordance with the third objective of the project.

150. If, after review of primer and sealant field evaluations, improvements are shown, it is recommended that additional field projects be undertaken. These projects would provide the data required to adopt the proposed specifications.

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APPENDIX A: SUMMARY OF SELECTED TEST PROCEDURES

APPENDIX A
SUMMARY OF SELECTED TEST PROCEDURES

Cone Penetration

1. The cone penetration is conducted very similarly to standard asphalt/cement penetration tests excepts that a special cone is substituted for the penetration needle (see Figure 1). There are two types of cone penetration test: nonimmersed penetration and fuel-immersed penetration.

2. In both the nonimmersed and fuel-immersed cone penetration tests a 6 oz (177 cubic centimeter) container is filled flush with the top edge of

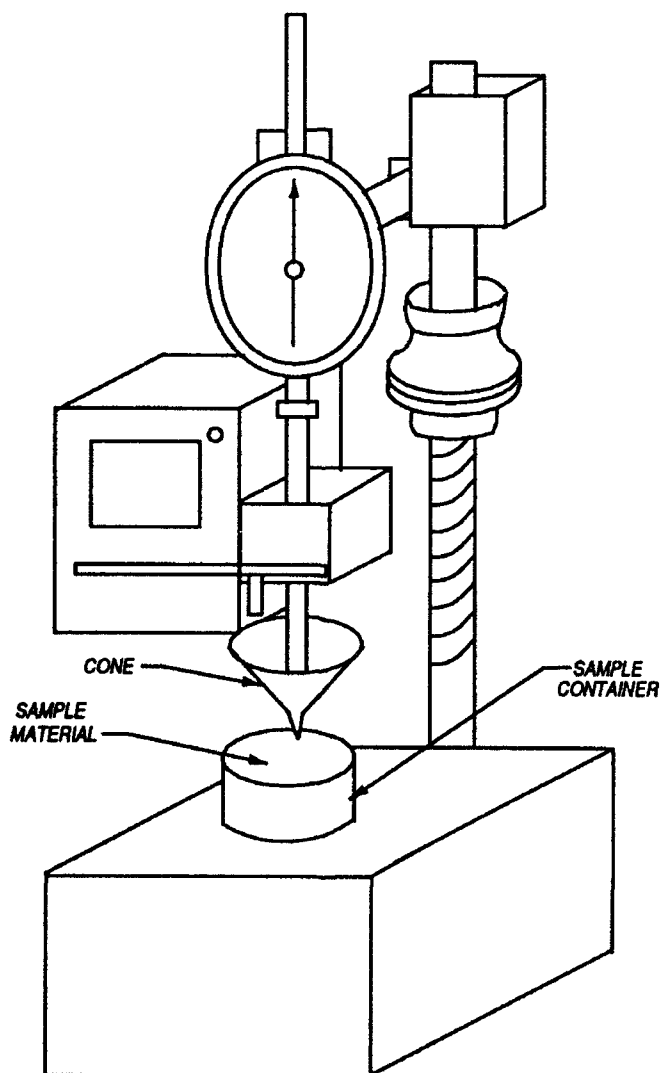


Figure 1. Cone penetrometer test setup

the container. The sample is placed on the penetrometer, the cone is brought to the sealant surface, released, and allowed to penetrate into the sample for 5 sec. The depth of penetration is measured by a dial on the penetrometer. The unit of measure is 1/10 mm and the total weight of the cone and penetrometer shaft used for testing is 150 gr.

3. The fuel-immersed penetration is conducted in the same manner except the sample is conditioned in ASTM D471 Reference Fuel B for 24 hr at 120°F. After conditioning, the sample is placed in front of a fan for 1 hr. The sample is then tested in the same manner as the nonimmersed. See FS SS-S-1614A and ASTM D5 for more detailed information.

Bond

4. For each bond test, three bond test specimens are prepared with the sealant being poured to fill the area as shown in Figure 2. After proper curing the specimens are placed in the bond test machine (Figure 3) to maintain a constant temperature with forced air circulation for not less than 4 hr. The standard FF-SS-S-1614A test was conducted which consisted of conditioning three bond specimens with spacers maintaining the 1/2 in. separation of the concrete blocks at $0^{\circ}\pm 2^{\circ}\text{F}$ for not less than 4 hr. The specimens were then extended 0.25 in. at a uniform rate of 1/8 in. per hour while maintaining the specimens at the test temperature. The specimens are removed from the extension machine and the 1/2 in. spacers reinserted to permit the specimen to attempt to return to the original dimensions at standard conditions. In addition testing conditions for other different sets of specimens for three cycles were (a) $0^{\circ}\pm 2^{\circ}\text{F}$, 100 percent extension, (b) $-20^{\circ}\pm 2^{\circ}\text{F}$, 50 percent extension, and (c) $-20^{\circ}\pm 2^{\circ}\text{F}$, 100 percent extension.

5. Non-immersed bond. The following requirements shall be met:

- a. Two of the three specimens shall exhibit no crack, separation, or other opening in the sealant, or between the sealant and the concrete blocks.
- b. The third specimen shall exhibit no crack, separation, or other opening in the sealant, or between the sealant and the concrete blocks exceeding 0.25 in. in depth, and shall exhibit no total area of bare concrete exposed on the face of either single concrete block exceeding 0.25 square in.

6. Fuel-immersed bond. The following requirements shall be met:

- a. None of the three specimens shall exhibit any crack, separation, or other opening in the sealant, or between the sealant and the concrete blocks exceeding 0.25 in. in depth.

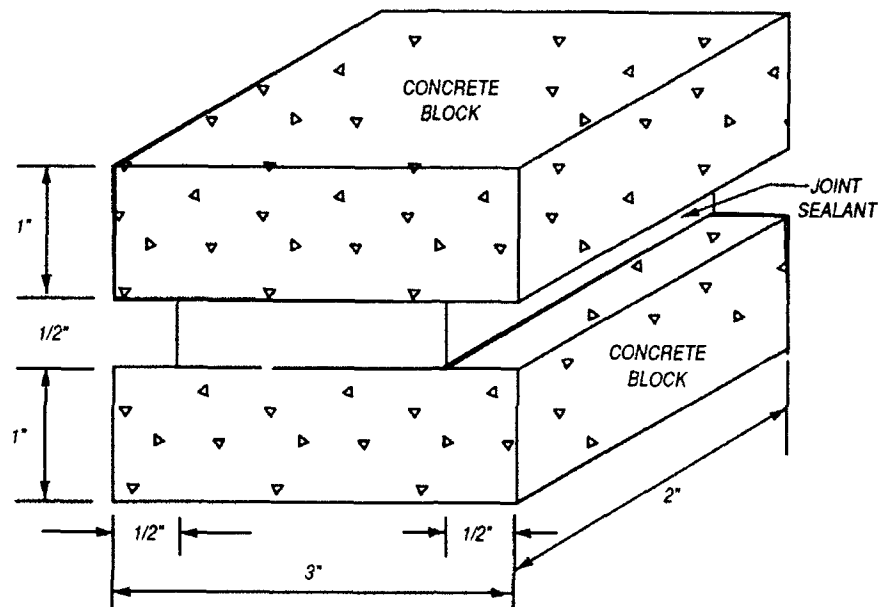


Figure 2. Bond test specimens

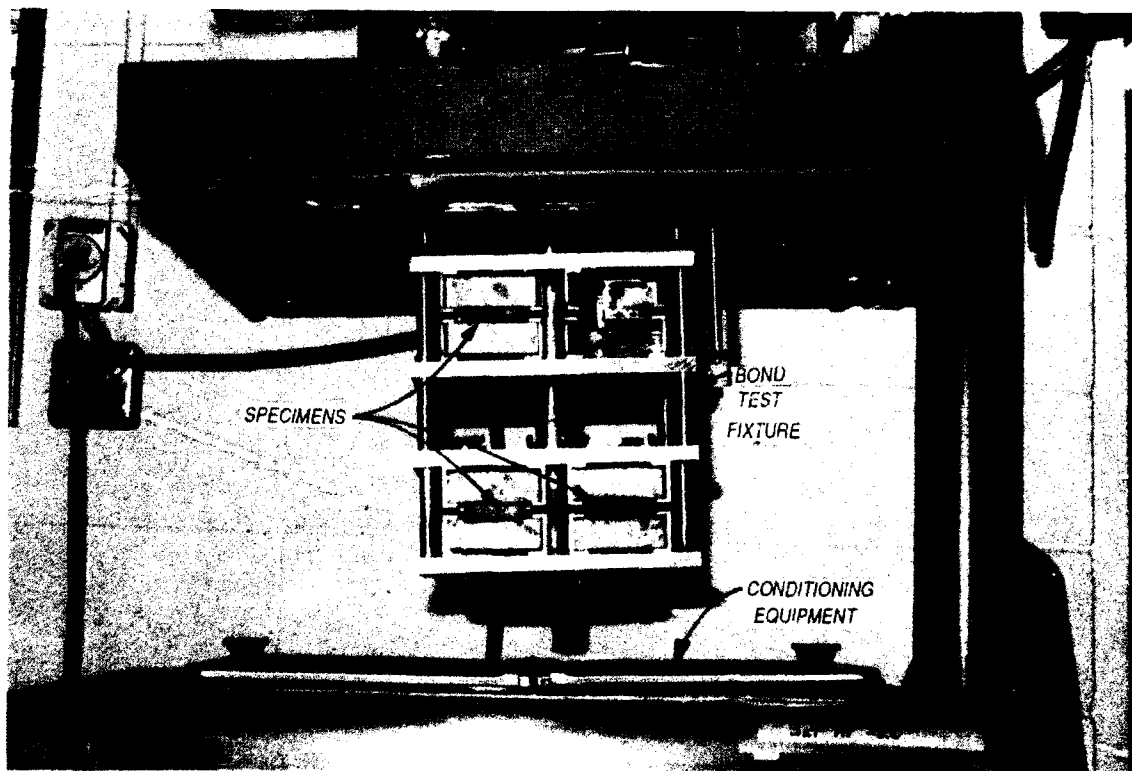


Figure 3. Cold temperature bond test machine

- b. None of the three specimens shall exhibit a total area of bare concrete exposed on the face of any single concrete block exceeding 0.25 square in.

7. Water-immersed bond. Requirements a., and b. of nonimmersed bond shall be met.

Ductility

8. The ductility of joint sealants is determined similar to the ductility determination of bituminous materials. A sample of joint sealant is poured and cured in the mold shown in Figure 4 when assembled on a flat brass plate. The specimen then is placed in the water at 77°F for 85 to 95 min. The clips of the mold are attached to the pins of the test machine (Figure 5) and the sides of the mold and brass plate are removed. The clips are then pulled at a uniform speed of 5 cm/min until the sample of joint sealant ruptures. The distance in centimeters through which the clips moved to produce rupture of the sample is measured and recorded. The specific gravity of the water bath is adjusted so that the sample of joint sealant does not come into contact with the bottom of the tank during the test. Three specimens are subjected to this test and the length to rupture is measured, averaged and recorded.

Flow

9. Two duplicate specimens are prepared for flow testing. Each specimen is prepared by placing a mold on a bright tin panel, pouring the sealant into the mold and striking the sealant flush with the top of the mold. The interior dimensions of the mold are 40 mm by 60 mm by 3.2 mm. The tin panel is used for the bottom of the mold.

10. The sealant is allowed to cool at standard laboratory temperature (73±4°F) for a minimum of 1/2 hr and then the mold is removed. A reference line is marked across the panel coincident with the transverse edges (40 mm) of the sealant. The specimen is then mounted into a specimen holder (Figure 6) that maintains it at a 75° angle with the horizontal. The specimen is mounted so that the 60 mm edges are vertical. The specimen holder is then placed in a forced-draft oven maintained at 140°F for 5 hr. After 5 hr the specimen holder is removed from the oven and the specimen is allowed to cool. A reference line is made coincident with the transverse edge of the sealant. The distance between the two reference lines is measured in mm and is reported as flow.

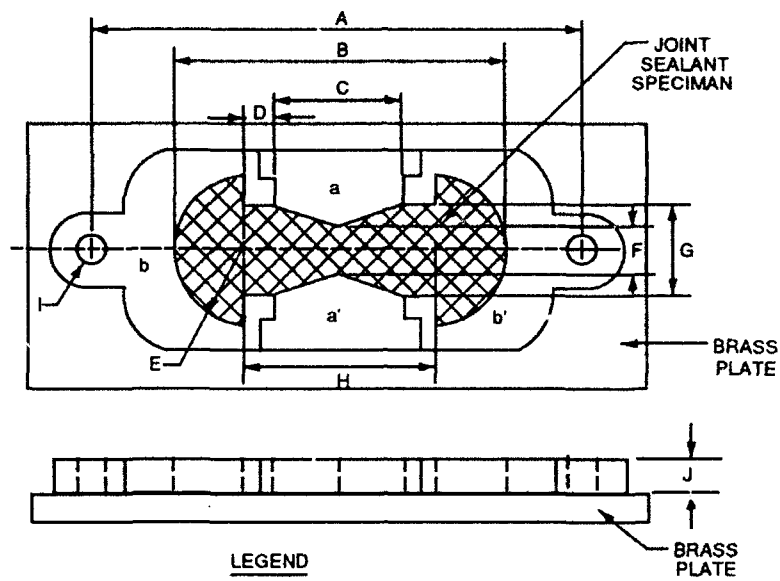


Figure 4. Mold for ductility test sample

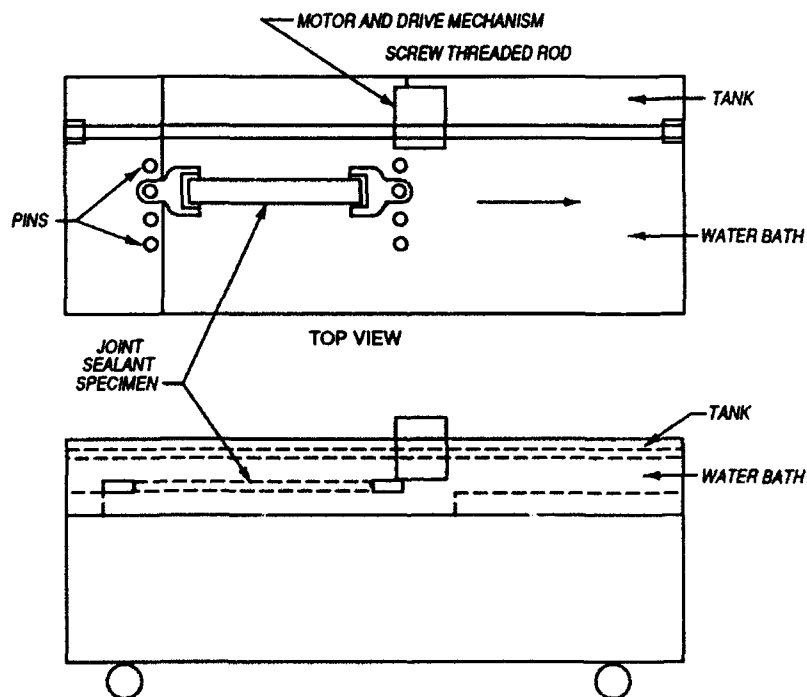


Figure 5. Ductility test machine

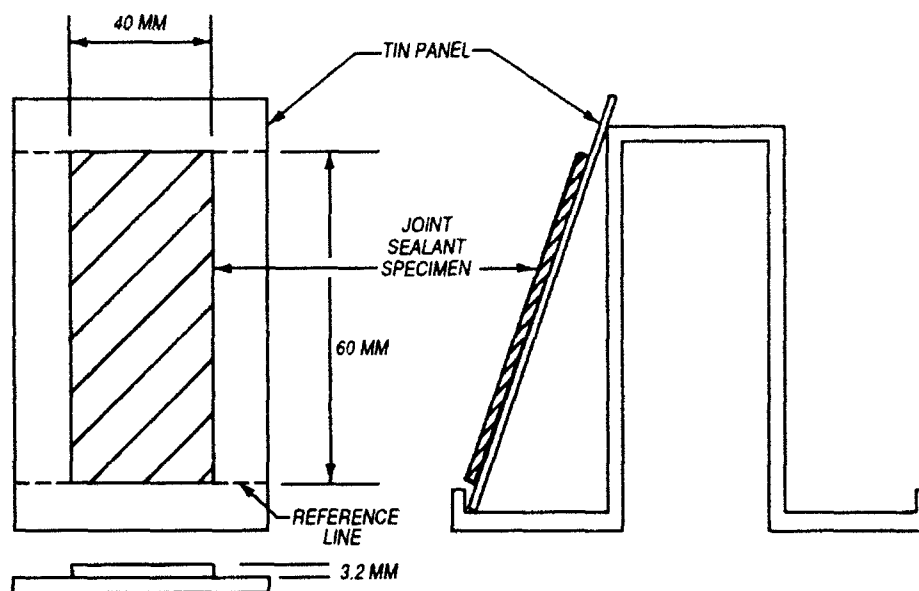


Figure 6. Flow test setup

Resilience

11. The test specimens are prepared as those for the cone penetration tests. Test apparatus is also the same as that for the penetration tests except a ball penetration tool (see Figure 7) is used instead of the needle.

12. Tests were performed in accordance with FS SS-S-1401C except four different curing and conditioning periods prior to conditioning in the 77°F water bath were used, (a) 24 ± 2 hr at standard conditions $73 \pm 4^\circ\text{F}$ and 50 ± 5 percent relative humidity (standard FS SS-S-1401C standard procedure), (b) 72 ± 4 hr at standard conditions (Standard ASTM D3583 procedure), (c) 72 ± 4 hr at standard conditions followed by 24 ± 2 hr in a $158 \pm 2^\circ\text{F}$ forced draft oven (Standard ASTM D3583 aged procedure), and (d) 72 ± 4 hr at standard conditions followed by 168 ± 4 hr in a $158 \pm 2^\circ\text{F}$ forced-draft oven (FF SS-S-1401C aged procedure). Each specimen was then allowed to cool at standard conditions for 1 hr then conditioned in a water bath maintained at $77 \pm 0.5^\circ\text{F}$ prior to testing.

13. The surface of the specimen is lightly dusted with talc and the excess is removed by blowing. The ball is placed in contact with the surface

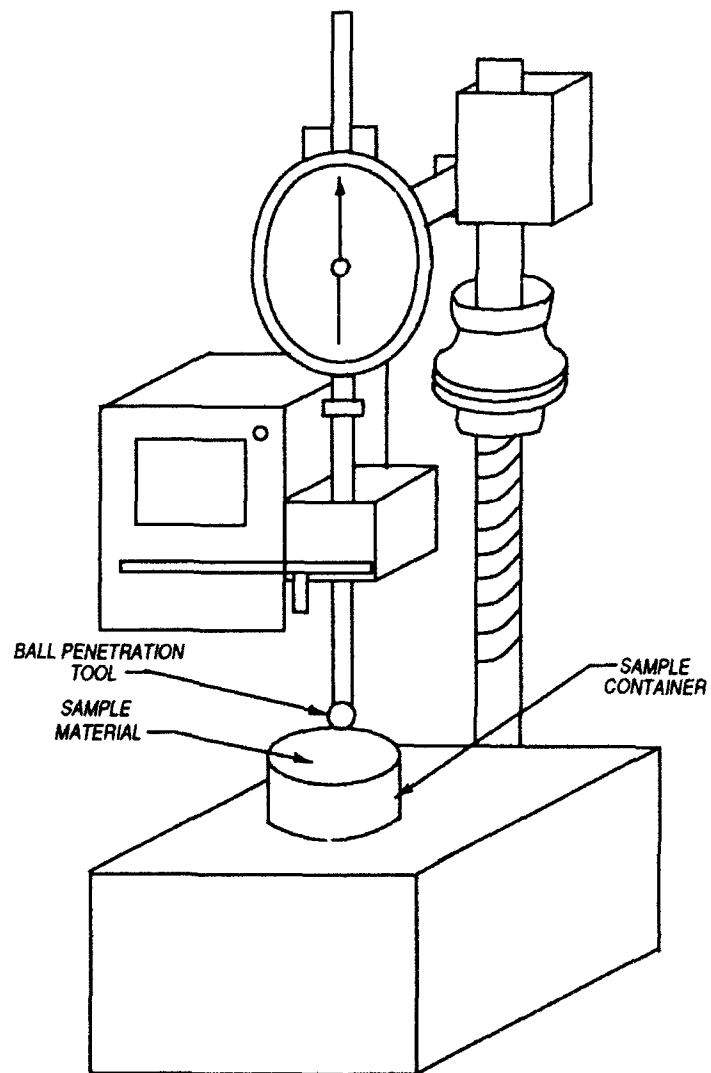


Figure 7. Resilience test setup

of the specimen and the indicating dial is set to zero. A light is positioned so that initial contact of the ball with the surface of the specimen can be observed readily. The ball penetration tool is released to allow it to penetrate the specimen for 5 sec. The reading as penetration (P) in tenth-millimeter units is recorded. Without returning the dial pointer to zero, the ball penetration tool is pressed down an additional 100 units (i.e., to a reading of $P + 100$) at a uniform rate in 10 sec. The clutch is re-engaged to hold the tool down for an additional 5 sec and during this time the dial is returned to zero. The clutch is released to allow the specimen to recover for 20 sec. The final dial reading (F) is recorded. Determinations are made at three points equally spaced from each other and not less than 13 mm (0.5 in.) from the container rim. The recovery, a measure of resilience, is calculated as follows.

$$\text{Recovery, percent} = P + 100 - F$$

Softening point

14. The softening point of joint sealants is determined similar to that of bitumen materials. Two disk of joint sealant, each 1.6 mm thick and 19.8 mm in diameter are poured and cured in a brass mold. These samples are then placed in a two-ring assembly test fixture (see Figure 8). A steel ball is placed in the center of each specimen and held in place with a centering guide. An 800-ml glass beaker with ethylene glycol to a level of $4 \frac{1}{8} \pm \frac{1}{8}$ in. is then used to contain the two-ring assembly with the joint sealant specimens. A thermometer is placed in the center of the two-ring assembly to monitor the temperature. The beaker is then heated to a point at which the weight of the ball on each joint sealant specimen causes the joint sealant to touch the plate 1 in. below each specimen. The temperature at which this occurs is recorded and this is the softening point of the joint sealant specimen.

Static adhesion

15. For the static adhesion test, three specimens are prepared as those for the bond test. See Figure 2 under "bond" for size etc. of specimens. The specimens are then placed in the test fixture (see Figure 9) that is put into the conditioning equipment and maintained at 0°F for a minimum of 4 hr. The

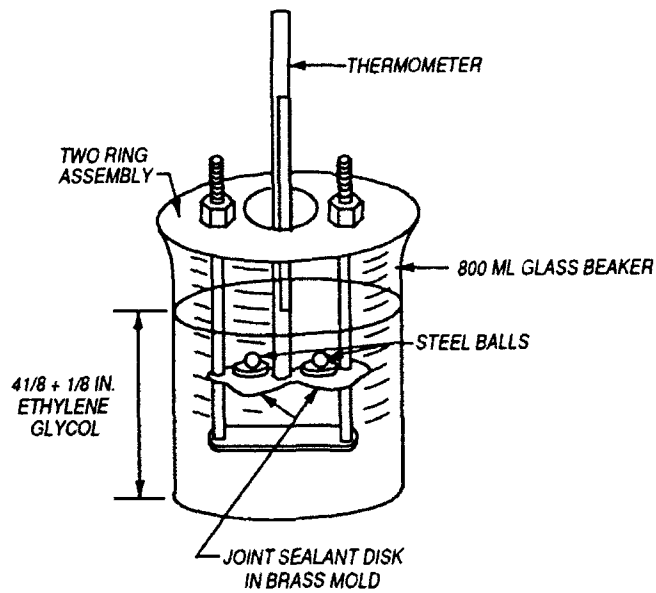


Figure 8. Joint sealant disks in brass mold

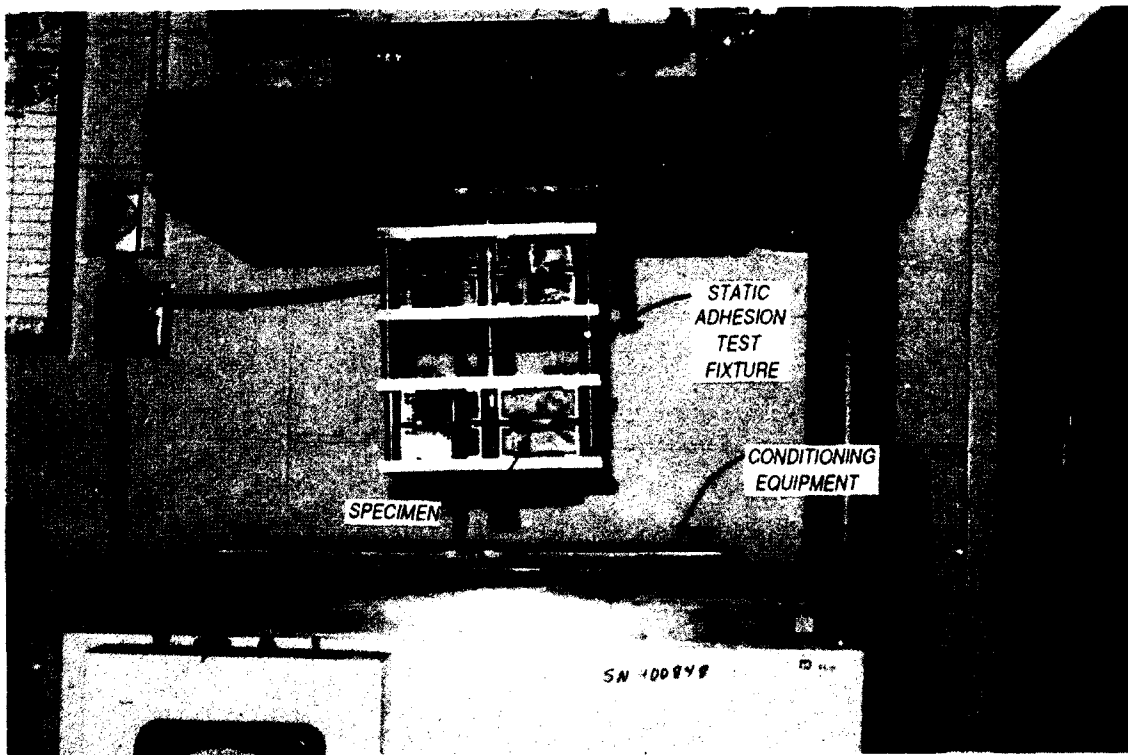


Figure 9. Static adhesion test setup

specimens inside the equipment are extended at 0°F to 100 percent extension of the specimen at a rate of 0.125 in. per hr (0.50 in extension). The specimens are then removed from the conditioning equipment, 1-in. thick spacers (Figure 10) are placed between the concrete blocks to maintain 100 percent extension. The specimens are then removed from the test fixture and maintained at 72°F for 24 hr. After this period the specimens are examined for any loss of adhesion to the concrete blocks. A passing result is one which has less than 160 mm² of adhesive separation.

Tensile adhesion

16. For each tensile adhesion test, three test specimens are prepared as specified in ASTM D3583 with the sealant being poured to fill the area shown in Figure 11. After proper curing, the specimens are subjected to tensile forces as shown in Figure 12. The concrete blocks are separated at a rate of 0.5 ± 0.1 in./min at 77°F. The extension is continued until cohesive or adhesive failure of the specimen occurs. The elongation at failure is recorded and averaged for the three specimens. Type of failure is noted and percent elongation is calculated of each specimen.

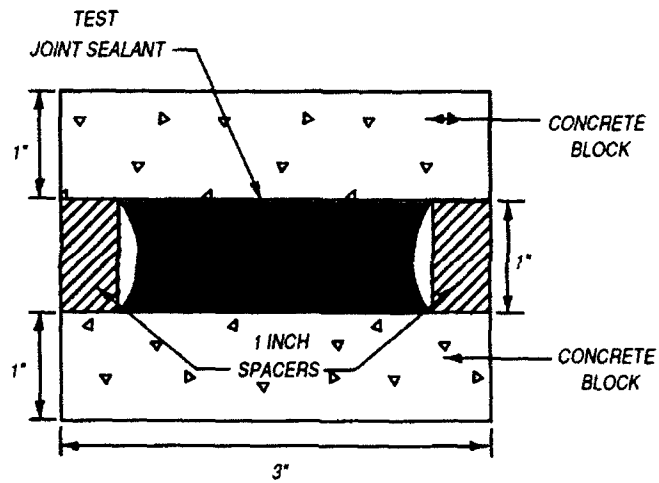


Figure 10. Static adhesion test specimen

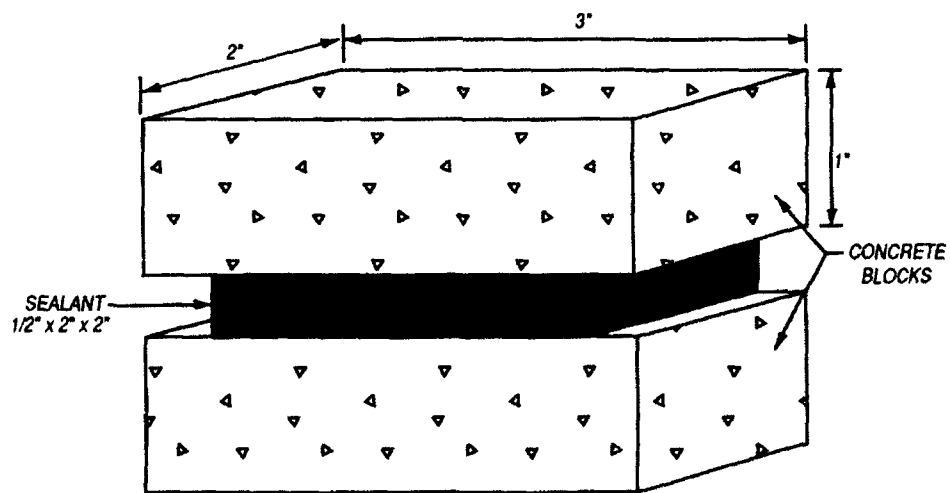


Figure 11. Tensile adhesion test sample

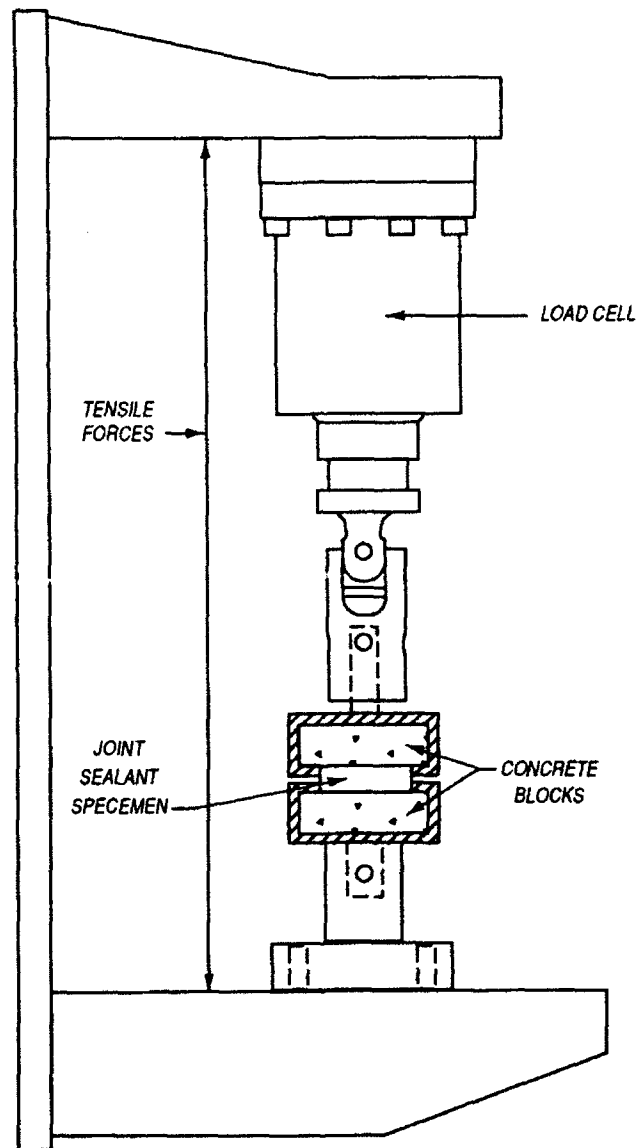


Figure 12. Test setup for tensile adhesion

APPENDIX B: PROPOSED SPECIFICATION FOR LOW-MODULUS, HOT-APPLIED,
JET-FUEL-RESISTANT PAVEMENT JOINT SEALANTS
FOR RIGID PAVEMENTS

NOTE: This proposal is not finalized and changes may occur before it is published as a material specification. Therefore, it may not be applicable for project specifications.

1. SCOPE: This specification covers low-modulus, hot-applied, jet-fuel-resistant pavement joint sealant materials for use in sealing joints and cracks in rigid pavements that are exposed to cold ambient temperatures.

2. APPLICABLE DOCUMENTS

2.1 The following documents, of the issues in effect on date of invitation for bids or request for proposal, form a part of this specification to the extent specified herein.

Federal Specification

PPP-P-704 - Pails, Metal: (Shipping, Steel, 1 Through 12 Gallons)

Federal Standards

FED-STD-123 - Marking for Shipment (Civil Agencies)

FED-STD-313 - Material Safety Data Sheets, Preparation and the
Submission of,

Military Standards

MIL-STD-105 - Sampling Procedures and Tables for Inspection by
Attributes

MIL-STD-129 - Marking for Shipment and Storage

MIL-STD-147 - Palletized Unit Loads

Federal Regulations

29 CFR 1900-1999 - Occupational Safety and Health Administration
(OSHA), Department of Labor

2.2 Other publications. The following documents form a part of this specification to the extent specified herein. Unless a specific issue is identified, the issue in effect on date of invitation for bid or request for proposal shall apply.

American Society for Testing and Materials (ASTM)

D5 - Penetration of Bituminous Materials, Test Method for
D140 - Sampling Bituminous Materials, Methods of
D217 - Cone Penetration of Lubricating Grease, Test Method for
D471 - Rubber Property - Effect of Liquids, Test Method for
D1985 - Preparing Concrete Blocks for Testing Sealants for
Joints and Cracks, Standard Practice for
D5167 - Melting of Hot-Applied Joint and Crack Sealant and Filler
Material for Evaluation, Standard Practice for

Technical Association of the Pulp and Paper Industry

T431 om - Ink Absorbency of Blotting Paper

2.3 Order of Precedence. In the event of a conflict between the text of this specification and the references cited herein, the text of this specification shall have precedence.

3. REQUIREMENTS

3.1 Description.

3.1.1 Material. The sealant shall be a thermoplastic material. It shall be composed of a mixture of coal tar and rubber or resin with additives. The manufacturer's specified application or pouring temperature shall not exceed

232 deg Celsius (°C) (450 deg Fahrenheit (°F)), and shall be at least 11°C (20°F) lower than the safe heating temperature. The sealant shall meet the requirements of this specification when poured after held at the application temperature for no more than 10 min, and shall retain all of its properties with up to 3 hr of continuous heat.

3.1.2 Performance. The sealant shall form a resilient and adhesive compound, resistant to the solvent action of jet fuels and lubricating oils. The sealant shall effectively seal joints and cracks in pavements against the infiltration of moisture throughout repeated cycles of expansion and contraction. The sealant shall not flow from the joint or be picked up by pneumatic tires at ambient temperatures of 52°C (125°F) or below. The sealant shall have a uniform application or pouring consistency suitable for filling the joint or crack without the inclusion of blisters, bubbles, or discontinuities.

3.2 Safe Heating Temperature. The safe heating temperature is the highest used temperature permitted by the manufacturer (see 5.3.3) and is a temperature to which the sealant can be heated for a duration of at least 3 hr, and still conform to all of the requirements specified herein.

3.3 Penetration.

3.3.1 Nonimmersed Penetration. The nonimmersed penetration shall not exceed 16.0 millimeters (mm) (160 test units) (0.630 in.) when the sealant is tested as specified in 4.4.3.1.

3.3.2 Fuel-Immersed Penetration. The fuel-immersed penetration shall not exceed the nonimmersed penetration when the sealant is tested as specified in 4.4.3.2.

3.3.3 Aged Penetration Retention. The aged penetration retention shall be a minimum of 70 percent when tested as specified in 4.4.3.3.

3.4 Change in Mass by Fuel Immersion. The change in dry mass after fuel immersion for 24 hr at 49°C (120°F) shall not exceed 1.0 percent, and there

shall be no apparent defects that will affect the material as a sealant when sealant is tested as specified in 4.4.4.

3.5 Flow. Flow shall not exceed 3.0 mm (0.118 in.) for either specimen when tested as specified in 4.4.5.

3.6 Bond to Concrete.

3.6.1 Nonimmersed Bond. When the sealant is tested as specified in 4.4.6.4, the following requirements shall be met:

a. Two of the three specimens shall exhibit no crack, separation, or other opening in the sealant, or between the sealant and the concrete blocks.

b. The third specimen shall exhibit no crack, separation, or other opening in the sealant, or between the sealant and the concrete blocks exceeding 6.35 mm (0.25 in.) in depth, and shall exhibit no total area of bare concrete exposed on the face of either single concrete block exceeding 160 mm² (0.25 in.²).

3.6.2 Fuel-Immersed Bond. When the sealant is tested as specified in 4.4.6.5, the following requirements shall be met:

a. None of the three specimens shall exhibit any crack, separation, or other opening in the sealant, or between the sealant and the concrete blocks exceeding 6.35 mm (0.25 in.) in depth.

b. None of the three specimens shall exhibit a total area of bare concrete exposed on the face of any single concrete block exceeding 160 mm² (0.25 in.²).

3.6.3 Water-Immersed Bond. When the sealant is tested as specified in 4.4.6.6, requirements a. and b. of 3.6.1 shall be met.

3.7 Resilience. Recovery shall be a minimum 45 percent when the sealant is tested as specified in 4.4.7.

3.8 Storage Stability. When specified (see 6.2), the user agency will retain samples for verification of these requirements: The sealant, when stored for 2 years from date of delivery, at temperatures from -18°C to 46°C (0°F to 115°F), and tested in accordance with this specification, shall meet all of the requirements herein.

3.9 Toxicity. The material shall have no adverse effect on the health of personnel when used for its intended purpose in the manner recommended by the manufacturer. Questions pertinent to this effect shall be referred by the acquiring activity to the appropriate medical service who will act as advisor to the acquiring activity. The manufacturer's instructions shall provide personnel protection to meet OSHA requirements, including 29 CFR 1910.1000, 1910.1002, and 1910.1017, as applicable (see 4.5).

3.10 Material Safety Data Sheets (MSDS). MSDSs shall be prepared in accordance with FED-STD-313 and submitted as directed (see 6.2, 6.3, and 6.5).

4. QUALITY ASSURANCE PROVISIONS

4.1 Responsibility for Inspection. Unless otherwise specified, the contractor is responsible for the performance of all inspection requirements and may use his own or any other facilities suitable for the performance of the inspection requirements that are approved by the user agency. The user agency reserves the right to perform any inspections set forth in the specification where such inspections are deemed necessary to assure supplies and services conform to prescribed requirements.

4.4.1 Materials Inspection. The contractor is responsible for insuring that supplies and materials are inspected for compliance with all the requirements specified herein and in applicable referenced documents.

4.2 Classification of Inspections. The inspection requirements specified herein are classified as follows:

- a. Quality conformance inspection (see 4.2.1)

b. Inspection of preparation for delivery (see 4.6)

4.2.1 Quality Conformance Inspection. The quality conformance inspection shall be as specified in 4.4. Sampling shall be in accordance with 4.3.

4.3 Sampling. Unless otherwise specified (see 6.2), samples for testing shall be taken at the point of manufacture in accordance with ASTM D 140. It shall be the responsibility of the contractor to determine that the samples taken are representative of the batches for shipment. The representative composite sample of the sealant shall consist of not less than 7 kg (15 lb) from each batch. A lot consisting of a single batch, if taken from filled containers, shall be sampled from 3 containers, selected at random, to make up the composite sample. Sample identification shall include the name of the testing agency, the contract or purchase order number, and special marking as specified in 5.3.3. Each container from which sample material has been taken shall be resealed and marked for identification.

4.4 Testing. Testing shall be conducted at a user-agency approved facility (see 6.2). Samples taken as specified in 4.3 shall be tested as specified in 4.4.1 through 4.4.7. Individual test values and results of failure analyses of individual shall be recorded. Failure of the sealant to pass any test shall be cause for rejection of the lot, except as noted for bond specimen casting defects in 4.4.6.3, and as noted for a marginal test result. The exceptions for marginal test results are as follows: where results of only one of the specified tests are judged by the testing agency to be marginal as to meeting requirements, the testing agency has the option to perform a retest. If the testing agency does not exercise its option to retest, either the contractor or the user agency may request that a retest for that property be made at the requester's expense. Such a test will be made only when an adequate quantity of the original sample is available or where additional material can be obtained from the previously marked sampled containers.

4.4.1 Standard Conditions. Laboratory atmospheric conditions, hereinafter referred to as standard conditions, shall be a temperature of $23 \pm 2^{\circ}\text{C}$ ($73 \pm 4^{\circ}\text{F}$) and 50 ± 5 percent relative humidity. Specimens shall be stored and tested at standard laboratory conditions unless otherwise specified.

4.4.2 Specimen Preparation.

4.4.2.1 Equipment. The unit for melting the laboratory samples shall be as specified in ASTM D5167.

4.4.2.2 Melting. The portion of the sample selected for testing shall provide approximately 1.6 cubic decimeter (1.7 quarts) of melted material. The portion of material selected shall be obtained from the original sample and melted as specified in ASTM D5167. The sample shall be heated at the safe heating temperature until 3 hr have elapsed since the first segment was added to the melters.

4.4.2.3 Pouring. Pour all test specimens as specified in 4.4.5 through 4.4.7 within a period of 10 min. Discard the first 50 to 60 grams of material discharged from each melting pot. After pouring, store all specimens at standard laboratory conditions for 72 ± 4 hr prior to beginning testing or initiating fuel or water immersion procedures.

4.4.3 Penetration.

4.4.3.1 Nonimmersed Penetration. Testing shall be as specified in ASTM D5 except as specified herein. Use a penetrometer as specified in ASTM D217 with a cone conforming to the optional cone therein in place of the needle. Prepare specimen in 177 cubic centimeter (cc) (6 oz) container, and cast flush with the top edge. Make determinations at locations on 120° radii, and half-way between the center and outside of the specimen. Determine conformance to the requirements of 3.3.1.

4.4.3.2 Fuel-Immersed Penetration. Immerse specimens prepared as specified in 4.4.3.1, for 24 hr in 500 cc (16.9 oz) each of clean test fuel maintained at $49 \pm 1^\circ\text{C}$ ($120 \pm 2^\circ\text{F}$). The container for the test fuel and specimens shall have a 3.2 mm (0.125 in.) round hole cut in the lid to eliminate pressure build-up. More than one specimen of the same manufacturer's material may be immersed in the same container, provided the volume of test fuel per specimen is maintained at 500 cc. The container shall be deep enough to provide a minimum cover of 12.7 mm (0.5 in.) over the specimens. Use a covered constant

temperature water bath to maintain the container, specimens and test fuel at the required temperature. The test fuel shall be a 70 percent isooctane/30 percent toluene composition, by volume, conforming to the requirements of ASTM Reference Fuel B of ASTM D471 (see 6.6.2). Immediately after the 24-hr immersion period, dry the specimens for 1 hr under a 300 mm (12 in.) diameter electric fan, placed to provide an air speed of 0.76 to 2.54 meters per sec (m/s) (150 to 500 ft per min (f/m)) over the surface of the specimens. Test as specified in 4.4.3.1. Determine conformance to the requirements of 3.3.2.

4.4.3.3 Aged Penetration Retention. Prepare one specimen as specified in 4.4.3.1. Cure the specimen at standard laboratory conditions for 72±4 hr and then place the specimen in a forced-draft oven maintained at 70±1°C (158±2°F) for 72±1 hr. Remove the specimen from the oven and cool at standard laboratory conditions for 1 hr. Further condition the specimen for 1 hr in a constant temperature water bath maintained at 25±0.1°C (77±0.2°F). Determine the aged penetration as specified in 4.4.3.1. Calculate the aged penetration retention using the following formula:

$$\frac{\text{Oven Aged Penetration}}{\text{Nonimmersed Penetration}} \times 100\%$$

Determine conformance to the requirement of 3.3.3.

4.4.4 Change in Mass by Fuel Immersion. Prepare a specimen as specified in 4.4.3.1, in a tared container, and determine the mass to the nearest 0.01 gram. Immerse and dry the specimen as specified in 4.4.3.2, and redetermine the mass. Report the change in mass of the specimen in percent gain or loss. Determine conformance to the requirements of 3.4.

4.4.5 Flow.

4.4.5.1 Specimen Preparation. Prepare duplicate specimens in molds with inside dimensions of 40 mm by 60 mm by 3.2 mm (1.56 in. by 2.34 in. by 0.125 in.) placed on a bright tin panel. Metal molds coated with a release agent, such as a thin, cured film of heat-stable silicone (see 6.6.3), or

equivalent, shall be used. Fill the molds with excess sealant and allow the specimens to cool at standard laboratory conditions for a minimum of 30 min. After the specimens have cooled, trim off the excess sealant flush with the face of the mold with a heated knife or spatula.

4.4.5.2 Test. Remove the molds and mark reference lines across the panels coincident with the transverse edges of the specimens. Mount the specimens with the long axis at an angle of $75 \pm 1^\circ$ with the horizontal and the transverse axis horizontal, in a forced-draft oven maintained at $60 \pm 1^\circ\text{C}$ ($140 \pm 2^\circ\text{F}$). After 5 hr, remove the specimens and mark a reference line on each specimen coincident with the lowest point of sag or flow, and parallel to the reference line. Measure the indicated change in length of the specimen and report as flow. Determine conformance to the requirements of 3.4.

4.4.6 Bond to Concrete.

4.4.6.1 Extension Machine. The extension machine used in the bond test shall be so designed that the specimen can be maintained at the test temperature while being extended at a uniform rate as specified. It shall consist essentially of one or more screws rotated by an electric motor through suitable gear reductions. Self-aligning plates or grips, one of each pair fixed and the other carried by the rotating screw or screws, shall be provided for holding the test specimen in position during the test.

4.4.6.2 Concrete Block Preparation. The concrete blocks used for bond testing shall be prepared as specified in ASTM D1985. The blocks may be prepared by the testing agency or procured (see 6.6.3).

4.4.6.3 Specimen Preparation. Prepare 6 bond test specimens (12 blocks) as follows: Remove blocks from the storage water individually, scrub the 50 mm by 75 mm (2 in. by 3 in.) faces lightly with a stiff bristle brush, under running water, and resubmerge in fresh tap water until all blocks have been scrubbed. Remove all blocks from the water and lightly blot with an oil-free, soft, absorbent cloth or paper to remove all free surface water. Place the blocks, 3 each, with 50 mm by 75 mm faces down, centered and uniformly spaced 25 mm (1 in.) apart on sheets of blotting paper placed on a plane, solid,

nonabsorbent surface. The sheets shall be approximately 100 mm by 240 mm (4 in. by 9.5 in.) cut from material having a maximum absorption time of 28 sec as measured by TAPPI T431 (see 6.6.6). At the end of 1 hr, assemble pairs of concrete blocks to provide test specimens. Complete setup and pour specimens within 1 hr. Spacers and the base plate used for the test specimens shall have nonadherent, nonreactive surfaces (see 4.4.5.1). Place spacer strips not less than 6.35 mm (0.25-in.) thick on a base plate to form an open space 12.7 mm (0.5 in.) wide and 50 mm (2 in.) long. Place pairs of the concrete blocks on the spacers so that the 25 mm by 75 mm faces are on the spacers and the 50 mm by 75 mm faces which were against the blotting paper form the space to be filled by the sealant. Space the blocks 12.7 ± 0.1 mm (0.500 ± 0.005 in.) apart with 12.7 ± 0.1 mm square by 75 mm long spacers. Corners may be slightly rounded, but discard spacers that have a diagonal dimension less than 16.51 mm (0.650 in.). Place these spacers at a distance from the ends of the blocks so that an opening 12.7 ± 0.1 by 50 mm by 50 mm (0.5 ± 0.005 in. by 2 in. by 2 in.) is formed. Place spacer strips that are a minimum of 12.7 mm thick on top of the blocks to provide for an overfill. Clamps or other suitable means may be used to hold the blocks and overfill spacers in position. Pour sealant prepared in accordance with 4.4.2 into the space between the blocks in sufficient quantity to bring it at least even with the top of the overfill spacers, and in a manner to exclude air pockets from being trapped in the sealant. After the specimens have cooled to standard conditions remove the excess sealant protruding beyond the top and bottom of the concrete blocks by trimming with a hot knife or spatula. If the material shrinks on cooling below the top of the blocks, or if other casting defects are apparent, discard the specimens and prepare additional ones. Cool the specimens at least 2 hr, but no more than 24 hr, at standard laboratory conditions before subjecting them to test conditions.

4.4.6.4 Nonimmersed Bond. Condition 3 bond test specimens with spacers maintaining the 12.7 mm dimensions at the test temperature of $-29 \pm 1^\circ\text{C}$ ($-20 \pm 2^\circ\text{F}$) with forced air circulation for 4 to 16 hr. Then extend the specimen 6.35 mm (0.25 in.) at a uniform rate of 3.18 mm (0.125 in.) per hr, while maintaining the specimens at the test temperature. Remove the specimens from the extension machine and reinsert the 12.7 mm spacers, and examine the spacers as described in 4.4.6.7. Then allow the specimens to return to the original

dimensions at standard conditions, resting each specimen on one concrete block so that the weight of the top block recompresses the joint sealant. Three cycles of conditioning, extension, and recovery shall be completed within 5 days after the start of the first cycle, and shall constitute one complete test for nonimmersed bond (see 4.4.6.7). When initiation of the second or third cycle is delayed, store the specimens at the test temperature.

4.4.6.5 Fuel-Immersed Bond. Insert thinner spacers between the concrete blocks of another three bond specimens, so that an opening of not less than 6.35 mm by 12.7 mm by 50 mm (0.25 in. by 0.5 in. by 2 in.) will be produced and maintained between the spacers and the sealant. Using the type containers and procedures described in 4.4.3.2, and the test fuel specified therein, immerse the specimens for 24 ± 0.25 hr in 500 cc (16.9 oz) of the test fuel maintained at $49 \pm 1^\circ\text{C}$ ($120 \pm 2^\circ\text{C}$) by means of a constant temperature water bath. Place the specimens with the concrete blocks in a horizontal position. Three specimens may be placed in the same container, provided that the fuel-to-specimen ratio is maintained. At the end of the 24 hr immersion period, condition the entire assembly of test specimens, fuel, and container in an atmosphere at $-29 \pm 1^\circ\text{C}$ ($-20 \pm 2^\circ\text{F}$) for 4 hr. Remove the test specimens from the fuel, remove the spacers, and conduct the extension test as specified in 4.4.6.4. One cycle of immersion and three cycles of conditioning, extension, and recovery shall constitute one complete test for water-immersed bond (see 4.4.6.6).

4.4.6.6 Water-Immersed Bond. Insert thinner spacers between the concrete blocks of the remaining three bond specimens, so that an opening of not less than 6.35 mm by 12.7 mm by 50 mm (0.25 in. by 0.5 in. by 2 in.) will be produced and maintained between the spacers and the sealant. Using covered containers deep enough to provide a minimum of 12.7 mm water cover, immerse the specimens for 96 hr in 500 cc (16.9 oz) of distilled or deionized water per specimen, and maintain at standard laboratory temperature. Place the specimens with the concrete blocks in a horizontal position. Three specimens may be placed in the same container, provided that the water-to-specimen ratio is maintained. At the end of the 96 hr immersion period, remove the test specimens from the water, remove the spacers, and remove the excess surface water from the specimens with a soft, dry absorbent material. Subject the specimens

to conditioning and extension test as specified in 4.4.6.4. One cycle of immersion and three cycles of conditioning, extension, and recovery shall constitute one complete test for water-immersed bond (see 4.4.6.7).

4.4.6.7 Bond Test Results. Remove the bond-test specimens from the extension machine within 30 min after the completion of the extension of each of the first two test cycles and examine the specimens for obvious separations within the sealant and between the sealant and the concrete blocks without distorting or manually causing extension of the specimens. Immediately upon completion of the final cycle, insert both sets of spacers or otherwise maintain 50 percent extension during examination and dimensional measurements, examining the specimens thoroughly, while still frozen, for separations between the sealant and concrete blocks and within the sealant, including surface cracks. This shall be accomplished without distorting the specimens, but after recovery as specified in 4.4.6.4, the specimens may be extended uniformly up to 25.4 mm (1 in.) to permit further detailed examination. Determine conformance to the requirements of 3.6.1, 3.6.2, and 3.6.3.

4.4.7 Resilience.

4.4.7.1 Specimen Preparation. Prepare one specimen as specified in 4.4.3.1 except cure the specimens for 72 ± 4 hr at standard laboratory conditions prior to testing. Condition the specimen for 1 hr in a water bath maintained at $25 \pm 0.03^\circ\text{C}$ ($77 \pm 0.5^\circ\text{F}$) prior to testing.

4.4.7.2 Procedure. Use a penetrometer as specified in ASTM D217, substituting the ball penetration tool illustrated in Figure 1 of ASTM D217 for the needle. Lightly dust the surface of the specimen with talc and immediately remove the excess by blowing. Place the ball in contact with the surface of the specimen and set the indicating dial to zero. Position a light so that initial contact of the ball with the specimen surface can be readily observed. Release the ball penetration tool, allow it to penetrate the specimen for 5 sec. Record this reading as penetration (P) in tenth-millimeter units. Without returning the dial pointer to zero, press the ball penetration tool down an additional 100 units (i.e., to a dial reading of $P+100$) at a uniform rate within 10 sec. Re-engage the clutch to hold the ball in this position

for 5 sec and during this time return the indicator dial to zero. Release the clutch, allow the specimen to recover for 20 sec, and record the final dial reading (F). Make determinations at 3 points equally spaced from each other and not less than 13 mm (0.5 in.) from the container rim. Calculate the recovery, a measure of resilience, as follows:

$$\text{Recovery, percent} = P + 100 - F$$

Report the average of 3 determinations of recovery for the specimen. Determine conformance to the requirements of 3.7.

4.5 Toxicological Data and Formulations. The manufacturer shall provide a listing of the components in the sealant that when heated could produce hazardous vapors (see 5.3.3). Where precautions need to be taken relative to the inhaling of, or skin and eye contact with the material or vapors, these precautions shall be included in the manufacturer's instructions (see 3.9 and 5.3.3.1).

4.6 Inspection of Preparation for Delivery.

4.6.1 Sampling. Sampling for inspection of filled containers shall be in accordance with MIL-STD-105, inspection Level II. The unit of product shall be one unit prepared for shipment.

4.6.2 Examination. Each filled container selected shall be inspected for conformance to the requirements of Section 5. Inspection shall be based on an Acceptable Quality level of 2.5 percent defective.

5. PREPARATION FOR DELIVERY

5.1 Packing. Packing shall be Level A, B, or commercial as specified (see 6.2).

5.1.1 Level A. The material shall be packed in a close-fitting, tapered 24-gage metal pail with gasket and lug cover. Pails shall have a wire handle securely attached to ears or clips which shall be attached to the body of the

pails. The exterior surfaces of the pails shall be coated as specified in PPP-P-704. The unit pack quantity shall be one unit of issue quantity specified in the contract or purchase order.

5.1.2 Level B. The material shall be packed the same as for Level A except that the exterior surfaces of the pail shall be coated with a commercial coating.

5.1.3 Commercial. The material shall be packed to insure carrier acceptance and safe delivery to the destination in containers complying with the rules and regulations applicable to the mode of transportation.

5.2 Palletization.

5.2.1 Level A. Unless otherwise specified (see 6.2), material shall be palletized in accordance with MIL-STD-147.

5.2.2 Level B and Commercial. When specified (see 6.2), material shall be palletized in accordance with MIL-STD-147.

5.3 Marking.

5.3.1 Civil Agencies. Shipments to civil agencies shall be marked in accordance with MIL-STD-123.

5.3.2 Military Agencies. Shipments to military agencies shall be marked in accordance with MIL-STD-129.

5.3.3 Special Marking. In addition to the marking of 5.3.1 or 5.3.2, and any special marking of the contract or order, the following information shall be shown on each pail:

- a. Name of sealant
- b. Specification number
- c. Manufacturer's name and material designation
- d. Manufacturer's lot and batch number

- e. Date of manufacturer (month and year)
- f. List of hazardous components (see 4.5)
- g. Quantity of sealant in pail (net weight)
- h. Application or pouring temperature
- i. Safe heating temperature
- j. Instructions for use

5.3.3.1 Instructions for Use. The instructions for use (see 6.7) shall include, but not limited to the following: ambient temperature and humidity ranges, and moisture conditions of joints, for successful installation; essential requirements for preparation of joints, heating of the sealant, handling, placing, and disposal of hot materials; and any restrictions to be adhered to in order to reduce hazards to personnel or to the environment. If it is not feasible to include all the instructions on the container without sacrificing legibility, the most important information shall be shown on the container and the full instructions referenced and furnished separately.

6. NOTES

6.1 Intended Use. This sealant is intended for sealing joints and cracks in pavements that are subjected to the spillage of jet fuels and lubricating oils. It is not intended to be resistant to the heat and blast of jet aircraft engines, except when aircraft are moving at moderate speeds.

6.2 Ordering Data. Purchasers shall select the preferred options permitted herein, and include the following in procurement documents:

- a. Title, number and date of this specification
- b. When stability samples are required, quantity to be retained and by what activity (see 3.7 and 6.4)
- c. Addresses for submission of MSDS (see 3.9 and 6.5)
- d. Sampling, if other than as specified (see 4.3)
- e. Designation of Government-approved test facility (see 4.4)
- f. Level of packing required (see 5.1)
- g. If palletization is not required for Level A (see 5.2.1)

- h. When palletization is required for Level B or commercial
(see 5.2.2)

6.3 Data Requirements. When this specification is used in an acquisition which incorporates DD Form 1423, Contract Data Requirements List (CDRL) and invokes the provisions of paragraph 52.227-7031 of the Federal Acquisition Regulations (FAR), the data requirements will be developed as specified by an approved Data Item Description (DD Form 1664) and delivered in accordance with the approved CDRL (DD Form 1423) incorporated into the contract. When the provisions are not invoked, the data shall be delivered in accordance with the contract requirements (see 3.10).

6.4 Stability Samples. The date of delivery shall be marked on samples submitted for stability testing (see 3.7).

6.5 MSDS Submission and Forwarding. MSDS copies shall be forwarded to the designated Industrial Hygienist and the focal point of the activity that purchased the item, and focal point of the using activity if different from purchasing activity. After review and acceptance of MSDS by designated recipients, approved copies will be forwarded to arrive at destinations prior to material delivery (see 3.9).

6.6 Availability of Testing Materials and Apparatus. Known suppliers of specified testing materials and apparatus are as follows:

6.6.1 Melting Unit. A unit as specified in 4.4.2.1: Laboratory Melter Model BLM-100, Berry Corporation, P.O. Box 337, Nicholasville, KY 40356.

6.6.2 Reference Fuel. Reference fuel as specified in 4.4.3.2: ASTM Reference Fuel B of ASTM D 471, Phillips Petroleum Company, Customer Service Center, Drawer "O", Borger, TX 79007.

6.6.3 Release Agent. An agent as specified in 4.4.5.1: Dow Corning 20 release coating, Dow Corning Corporation, Midland, MI 48640.

6.6.4 Concrete Blocks. Blocks as specified in 4.4.6.2: US Army Corps of Engineers, Missouri River Division Laboratory, 420 South 18th Street, Omaha, NE 68102.

6.6.6 Blotting Paper. Paper as specified in 4.4.6.3: White Reliance Blotting Paper, Product Code 13-01-12, James River Paper Company, Inc., 145 James Way, Southampton, PA 18966.

6.7 Precautions. The sealant material can be damaged by heating at too high a temperature, reheating, or by heating for too long a period of time. The temperature of the sealant in the melting equipment should never exceed the safe heating temperature set by the manufacturer. Any given quantity of material should never be heated at the application or pouring temperature for more than 3 hr and should never be reheated. Sealant material left in the equipment at the end of the working day should be removed and discarded. The sealant should be heated in a kettle or tank constructed as a double boiler, with the space between the inner and outer shells filled with heat transfer oil. Positive thermostatic control, mechanical agitation, and recirculating pumps should be provided to maintain a uniform temperature of the sealant and heating oil. Direct heating is not permitted. Thermometers should be provided for continuous temperature readings of both oil and the sealant.

APPENDIX C: PROPOSED SPECIFICATION FOR LOW-MODULUS, HOT-APPLIED,
NON-FUEL-RESISTANT, PAVEMENT JOINT SEALANTS
FOR RIGID AND FLEXIBLE PAVEMENTS

NOTE: This proposal is not finalized and changes may occur before it is published as a material specification. Therefore, it may not be applicable for project specifications.

1. SCOPE: This specification covers low-modulus, hot-applied, non-fuel-resistant pavement joint sealant materials for use in sealing joints and cracks in rigid and flexible pavements that are exposed to cold ambient temperatures.

2. APPLICABLE DOCUMENTS

2.1 The following documents, of the issues in effect on date of invitation for bids or request for proposal, form a part of this specification to the extent specified herein.

Federal Specification

PPP-P-704 - Pails, Metal: (Shipping, Steel, 1 Through 12 Gallons)

Federal Standards

FED-STD-123 - Marking for Shipment (Civil Agencies)

FED-STD-313 - Material Safety Data Sheets Preparation and the
Submission of

Military Standards

MIL-STD-105 - Sampling Procedures and Tables for Inspection by
Attributes

MIL-STD-129 - Marking for Shipment and Storage

MIL-STD-147 - Palletized Unit Loads

Federal Regulations

29 CFR 1900-1999 - Occupational Safety and Health Administration
(OSHA), Department of Labor

2.2 Other publications. The following documents form a part of this specification to the extent specified herein. Unless a specific issue is identified, the issue in effect on date of invitation for bid or request for proposal shall apply.

American Society for Testing and Materials (ASTM)

D5 - Penetration of Bituminous Materials, Test Method for
D140 - Sampling Bituminous Materials, Methods of
D217 - Cone Penetration of Lubricating Grease, Test Methods for
D1985 - Preparing Concrete Blocks for Testing Sealants for Joints
and Cracks, Standard Practice for
D5167 - Melting of Hot-Applied Joint and Crack Sealant and Filler
Material for Evaluation, Standard Practice for

Technical Association of the Pulp and Paper Industry

T431 om - Ink Absorbency of Blotting Paper

2.3 Order of Precedence. In the event of a conflict between the text of this specification and the references cited herein, the text of this specification shall have precedence.

3. REQUIREMENTS

3.1 Description.

3.1.1 Material. The sealant shall be a fully polymerized and cured thermoplastic material furnished as a solid. It shall be composed of a mixture of asphalt and rubber with additives. The manufacturer's specified application or pouring temperature shall not exceed 232 deg Celsius (°C) (450 deg

Fahrenheit (°F)), and shall be at least 11°C (20°F) lower than the safe heating temperature. The sealant shall meet the requirements of this specification when poured after held at the application temperature for no more than 10 min, and shall retain all of its properties with up to 3 hr of continuous heat.

3.1.2 Performance. The sealant shall form a resilient and adhesive compound, which effectively seals joints and cracks in pavements against the infiltration of moisture throughout repeated cycles of expansion and contraction. The sealant shall not flow from the joint or be picked up by pneumatic tires at ambient temperatures of 52°C (125°F) or below. The sealant shall have a uniform application or pouring consistency suitable for filling the joint or crack without the inclusion of blisters, bubbles, or discontinuities.

3.2 Safe Heating Temperature. The safe heating temperature is the highest use temperature permitted by the manufacturer (see 5.3.3) and is a temperature to which the sealant can be heated for a duration of at least 3 hr, and still conform to all of the requirements specified herein.

3.3 Penetration. Penetration shall not exceed 15.0 millimeters (mm) (150 test units) (0.591 in.) or be less than 9.0 mm (90 test units) (0.354 in.) when the sealant is tested as specified in 4.4.3.

3.4 Flow. Flow shall not exceed 3.0 mm (0.118 in.) for either specimen when tested as specified in 4.4.4.

3.5 Resilience. Recovery shall be a minimum of 50 percent and shall not exceed 80 percent when the sealant is tested as specified in 4.4.5. The initial indentation shall not exceed 3.5 mm (0.138 in.) or be less than 1.0 mm (0.039 in.) when tested as specified in 4.5.5.

3.6 Bond to Concrete. No specimen shall develop any surface crack, separation, or other opening in the sealant, or between the sealant and the concrete blocks when the sealant is tested as specified in 4.4.6.

3.7 Static Adhesion. When the sealant is tested as specified in 4.4.7, none of the 3 specimens shall exhibit any crack, separation, or other opening in the sealant, or between the sealant and the concrete blocks exceeding 6.35 mm (0.25 in.) in depth. Additionally, none of the 3 specimens shall exhibit a total area of bare concrete exposed on the face of any single concrete block exceeding 160 mm² (0.25 in.²).

3.8 Compatibility with Asphalt. There shall be no failure in adhesion, formation of an oily exudate at the interface between the sealant and the asphaltic concrete, or softening or other deleterious effect on the asphaltic concrete, when tested as specified in 4.4.7.

3.9 Storage Stability. When specified (see 6.2), the user agency will retain samples for verification of these requirements: The sealant, when stored for 2 years from date of delivery, at temperatures from -18°C to 46°C (0°F to 115°F), and tested in accordance with this specification, shall meet all of the requirements herein.

3.10 Toxicity. The material shall have no adverse effect on the health of personnel when used for its intended purpose in the manner recommended by the manufacturer. Questions pertinent to this effect shall be referred by the acquiring activity to the appropriate medical service who will act as advisor to the acquiring activity. The manufacturer's instructions shall provide personnel protection to meet OSHA requirements, including 29 CFR 1910.1000, 1910.1002, and 1910.1017, as applicable (see 4.5).

3.11 Material Safety Data Sheets (MSDS). MSDSs shall be prepared in accordance with FED-STD-313 and submitted as directed (see 6.2, 6.3, and 6.5).

4. QUALITY ASSURANCE PROVISIONS

4.1 Responsibility for Inspection. Unless otherwise specified, the contractor is responsible for the performance of all inspection requirements and may use his own or any other facilities suitable for the performance of the inspection requirements that are approved by the user agency. The user agency reserves the right to perform any inspections set forth in the specification

where such inspections are deemed necessary to assure supplies and services conform to prescribed requirements.

4.1.1 Materials Inspection. The contractor is responsible for insuring that supplies and materials are inspected for compliance with all the requirements specified herein and in applicable referenced documents.

4.2 Classification of Inspections. The inspection requirements specified herein are classified as follows:

- a. Quality conformance inspection (see 4.2.1).
- b. Inspection of preparation for delivery (see 4.6).

4.2.1 Quality Conformance Inspection. The quality conformance inspection shall be as specified in 4.4. Sampling shall be in accordance with 4.3.

4.3 Sampling. Unless otherwise specified (see 6.2), samples for testing shall be taken at the point of manufacture in accordance with ASTM D140. It shall be the responsibility of the contractor to determine that the samples taken are representative of the batches for shipment. The representative composite sample of the sealant shall consist of not less than 7 kilograms (15 lbs) from each batch. A lot consisting of a single batch, if taken from filled containers, shall be sampled from 3 containers, selected at random, to make up the composite sample. Sample identification shall include the name of the testing agency, contract or purchase order number, and special marking as specified in 5.3.3. Each container from which sample material has been taken shall be resealed and marked for identification.

4.4 Testing. Testing shall be conducted at a user-agency approved facility (see 6.2). Samples taken as specified in 4.3 shall be tested as specified in 4.4.1 through 4.4.7. Individual test values and results of failure analyses of individual shall be recorded. Failure of the sealant to pass any test shall be cause for rejection of the lot, except as noted for bond specimen casting defects in 4.4.6.3, and as noted for a marginal test result. The exceptions for marginal test results are as follows: where results of only

one of the specified tests are judged by the testing agency to be marginal as to meeting the requirements, the testing agency has the option to perform a retest. If the testing agency does not exercise its option to retest, either the contractor or the user agency may request that a retest for that property be made at the requester's expense. Such a test will be made only when an adequate quantity of the original sample is available or where additional material can be obtained from the previously marked sample containers.

4.4.1 Standard Conditions. Laboratory atmospheric conditions, hereinafter referred to as standard conditions, shall be a temperature of $23\pm 2^{\circ}\text{C}$ ($73\pm 4^{\circ}\text{F}$) and 50 ± 5 percent relative humidity. Specimens shall be stored and tested at standard laboratory conditions unless otherwise specified.

4.4.2 Specimen Preparation.

4.4.2.1 Equipment. The unit for melting the laboratory samples shall be as specified in ASTM D5167.

4.4.2.2 Melting. The portion of the sample selected for testing shall provide approximately 1.6 cubic decimeter (1.7 quarts) of melted material. The portion of material selected shall be obtained from the original sample and melted as specified in ASTM D5167. The sample shall be heated at the safe heating temperature until 3 hr have elapsed since the first segment was added to the melters.

4.4.2.3 Pouring. Pour all test specimens as specified in 4.4.3 through 4.4.7 within a period of 10 min. Discard the first 50 to 60 grams of material discharged from each melting pot.

4.4.3 Penetration. Penetration testing shall be as specified in ASTM D5 except as specified herein. Use a penetrometer as specified in ASTM D217 with a cone conforming to the optional cone therein in place of the needle. Prepare specimen in 177 cubic centimeter (cc) (6 oz) container, and cast flush with the top edge. Make determinations at locations on 120° radii, and half-way between the center and outside of the specimen. Determine conformance to the requirements of 3.3.

4.4.4 Flow.

4.4.4.1 Specimen Preparation. Prepare duplicate specimens in molds with inside dimensions of 40 mm by 60 mm by 3.2 mm (1.56 in. by 2.34 in. by 0.125 in.), placed on a bright tin panel. Metal molds, coated with a release agent, such as a thin, cured film of heat-stable silicone (see 6.6.3), or equivalent, shall be used. Fill the molds with excess sealant and allow the specimens to cool at standard laboratory conditions for a minimum of 30 min. After the specimens have cooled, trim off the excess sealant flush with the face of the mold with a heated knife or spatula.

4.4.4.2 Test. Remove the molds and mark reference lines across the panels coincident with the transverse edges of the specimens. Mount the specimens with the long axis at an angle of $75 \pm 1^\circ$ with the horizontal and the transverse axis horizontal in a forced-draft oven maintained at $60 \pm 1^\circ\text{C}$ ($140 \pm 2^\circ\text{F}$). After 5 hr, remove the specimens and mark a reference line on each specimen coincident with the lowest point of sag or flow, and parallel to the reference line. Measure the indicated change in length of the specimen and report as flow. Determine conformance to the requirements of 3.4.

4.4.5 Resilience.

4.4.5.1 Specimen Preparation. Prepare duplicate specimens as specified in 4.4.3, except cure the specimens for 24 hr at standard laboratory conditions prior to testing. Oven-age one specimen in a forced-draft oven at $70 \pm 1^\circ\text{C}$ ($158 \pm 2^\circ\text{F}$) for 168 ± 2 hr, cool at standard laboratory conditions for 1 hr, and then condition for 1 hr in a water bath maintained at $25 \pm 0.3^\circ\text{C}$ ($77 \pm 0.5^\circ\text{F}$) prior to testing. Condition the unaged specimen for 1 hr in a water bath maintained at $25 \pm 0.3^\circ\text{C}$ ($77 \pm 0.5^\circ\text{F}$) prior to testing.

4.4.5.2 Procedure. Use a penetrometer as specified in ASTM D217, substituting the ball penetration tool illustrated in Figure 1 for the needle. Lightly dust the surface of the specimen with talc and immediately remove the excess by blowing. Place the ball in contact with the surface of the specimen and set the indicating dial to zero. Position a light so that initial contact of the ball with the specimen surface can be readily observed. Release the ball

penetration tool and allow the ball to penetrate the specimen for 5 sec. Record this reading as penetration (P) in tenth-millimeter units. Without returning the dial pointer to zero, press the ball penetration tool down for an additional 100 units (i.e., to a dial reading of P+100) at a uniform rate within 10 sec. Re-engage the clutch to hold the ball in this position for 5 sec and during this time return the indicator dial to zero. Release the clutch, allow the specimen to recover for 20 sec, and record the final dial reading (F). Make determinations at 3 points equally spaced from each other and not less than 13 mm (0.5 in.) from the container rim. Calculate the recovery, a measure of resilience, as follows:

$$\text{Recovery, percent} = P + 100 - F$$

Report the averages of 3 determinations of recovery and initial penetration for each specimen. Determine conformance to the requirements of 3.5.

4.4.6 Bond to Concrete.

4.4.6.1 Extension Machine. The extension machine used in the bond testing shall be so designed that the specimen can be maintained at the test temperature while being extended at a uniform rate as specified. It shall consist essentially of one or more screws rotated by an electric motor through suitable gear reductions. Self-aligning plates or grips, one of each pair fixed and the other carried by the rotating screw or screws, shall be provided for holding the test specimen in position during the test.

4.4.6.2 Concrete Block Preparation. The concrete blocks used for bond testing shall be prepared as specified in ASTM D1985. The blocks may be prepared by the testing agency or procured (see 6.6.3).

4.4.6.3 Specimen Preparation. Prepare 6 bond test specimens (12 blocks) as follows: Remove blocks from the storage water individually, scrub the 50 mm by 75 mm (2 in. by 3 in.) faces lightly with a stiff bristle brush under running water, and resubmerge in fresh tap water until all blocks have been scrubbed. Remove all blocks from the water and lightly blot with an oil-free, soft, absorbent cloth or paper to remove all free surface water. Place the

blocks, 3 each, with the 50 mm by 75 mm faces down, centered and uniformly spaced 25 mm (1 in.) apart on sheets of blotting paper placed on a plane, solid, nonabsorbent surface. The sheets shall be approximately 100 mm by 240 mm (4 in. by 9.5 in.), cut from material having a maximum absorption time of 28 sec as measured by TAPPI T431 (see 6.6.4). At the end of 1 hr, assemble pairs of concrete blocks to provide test specimens. Complete setup and pour specimens within 1 hr. Spacers and the base plate used for the test specimens shall have nonadherent, nonreactive surfaces (see 4.4.4.1). Place the spacer strips not less than 6.35 mm (0.25 in.) thick on a base plate to form an open space 12.7 mm (0.5 in.) wide and 50 mm (2 in.) long. Place pairs of the concrete blocks on the spacers so that the 25 mm by 75 mm faces are on the spacers and the 50 mm by 75 mm faces which were against the blotting paper form the space to be filled by the sealant. Space the blocks 12.7 ± 0.1 mm (0.500 ± 0.005 in.) apart with 12.7 ± 0.1 mm square by 75 mm long spacers. Corners may be slightly rounded, but discard spacers that have a diagonal dimension less than 16.51 mm (0.650 in.). Place these spacer strips at a distance from the ends of the blocks so that an opening 12.7 ± 0.1 by 50 mm by 50 mm (0.5 ± 0.005 in. by 2 in. by 2 in.) is formed. Place spacer strips that are a minimum of 12.7 mm thick on top of the blocks to provide for an overfill. Clamps or other suitable means may be used to hold the blocks and overfill spacers in position. Pour sealant prepared in accordance with 4.4.2 into the space between the blocks in sufficient quantity to bring it at least even with the top of the overfill spacers, and in a manner essentially to exclude air pockets from being trapped in the sealant. After the specimens have cooled to standard conditions, remove the excess sealant protruding beyond the top and bottom of the concrete blocks by trimming with a hot knife or spatula. If the material shrinks on cooling below the top of the blocks, or if other casting defects are apparent, discard the specimens and prepare additional ones. Cool the specimens at least 2 hr, but no more than 24 hr at standard laboratory conditions before subjecting them to test conditions.

4.4.6.4 Nonimmersed Bond. Condition 3 bond test specimens, with spacers maintaining the 12.7 mm dimensions, at the test temperature of $-29 \pm 1^{\circ}\text{C}$ ($-20 \pm 2^{\circ}\text{F}$), with forced air circulation for 4 to 16 hr. Then extend the specimen 25.4 mm (1 in.) at a uniform rate of 3.18 mm (0.125 in.) per hr while maintaining the specimens at the test temperature. Remove the specimens from

the extension machine and reinsert the 12.7 mm spacers, and examine the spacers as described in 4.4.6.6. Then allow the specimens to return to the original dimensions at standard conditions, resting each specimen on one concrete block so that the weight of the top block recompresses the joint sealant. Three cycles of conditioning, extension, and recovery shall be completed within 5 days after the start of the first cycle, and shall constitute one complete test for nonimmersed bond. When initiation of the second or third cycle is delayed, store the specimens at the test temperature.

4.4.6.5 Water-Immersed Bond. Insert thinner spacers between the concrete blocks of the remaining three bond specimens so that an opening of not less than 6.35 mm by 12.7 mm by 50 mm (0.25 in. by 0.5 in. by 2 in.) will be produced and maintained between the spacers and the sealant. Using covered containers deep enough to provide a minimum of 12.7 mm water cover, immerse the specimens for 96 hr in 500 cc (16.9 oz) of distilled or deionized water per specimen, and maintain at standard laboratory temperature. Place the specimens with the concrete blocks in a horizontal position. Three specimens may be placed in the same container, provided that the water-to-specimen ratio is maintained. At the end of the 96 hr immersion period, remove the test specimens from the water, remove the spacers, and remove the excess surface water from the specimens with soft, dry, absorbent material. Subject the specimens to conditioning and extension test as specified in 4.4.6.4. One cycle of immersion and three cycles of conditioning, extension, and recovery shall constitute one complete test for water-immersed bond (see 4.4.6.6).

4.4.6.6 Bond Test Results. Remove the bond test specimens from the extension machine within 30 min after the completion of the extension of each of the first two test cycles and examine the specimens for obvious separations within the sealant and between the sealant and the concrete blocks without distorting or manually causing extension of the specimens. Immediately upon completion of the final cycle, insert both sets of spacers or otherwise maintain 50 percent extension during examination and dimensional measurements, examining the specimens thoroughly, while still frozen, for separations between the sealant and concrete blocks and within the sealant, including surface cracks. This shall be accomplished without distorting the specimens, but after recovery as specified in 4.4.6.4, the specimens may be extended uniformly up to 25.4 mm

(1 in.) to permit further detailed examination. Determine conformance to the requirements of 3.6.

4.4.7 Static Adhesion.

4.4.7.1 Specimen Preparation. Prepare 3 bond test specimens as specified in 4.4.6.2 and 4.4.6.3 of this specification.

4.4.7.2 Procedure. Condition 3 bond test specimens with spacers maintaining the 12.7 mm (0.5 in.) dimensions at the test temperature of $-29 \pm 1^{\circ}\text{C}$ ($-20 \pm 2^{\circ}\text{F}$) with forced air circulation for not less than 4 hr. Then extend the specimens 12.7 mm (0.5 in.) at a uniform rate of 3.18 mm (0.125 in.) per hr while maintaining the specimens at the test temperature. Remove the test specimens from the extension machine and insert 2.54 mm (1 in.) spacers to maintain the 12.7 mm extension. Store the specimens at standard laboratory conditions for 24 hr and then examine them for the separations between the sealant and the concrete blocks or within the sealant. Determine conformance to the requirements of 3.7.

4.4.8 Compatibility with Asphalt.

4.4.8.1 Specimen Preparation. Prepare duplicate specimens of hot-mix asphaltic concrete, not less than 100 mm (4 in.) in diameter and 64 mm (2.5 in.) high using 85 - 100 penetration asphalt cement. Density and asphalt content shall be for asphaltic concrete pavement mix design and design method as specified (see 6.2 and 6.7). Specimens other than circular, but of similar dimensions and properties may be used. Allow the specimens to cool to standard laboratory temperature, then cut a groove 100 mm (4 in.) long by 13 ± 3.2 mm (0.5 ± 0.125 in.) wide by 19 ± 3.2 mm (0.75 ± 0.125 in.) deep in the surface of the specimen by wet sawing with a power driven masonry saw. Remove all residue from the grooves by scrubbing with a stiff-bristle brush under running water. Allow the specimens to return to standard laboratory conditions and securely wrap with cloth-backed adhesive tape, or otherwise reinforce to prevent slumping during the test period. Caulk the ends of the grooves to prevent leaking of the sealant during testing. Pour sealant, prepared in accordance with 4.4.2, into the groove, overfilling slightly, but not allowing overflow onto

the adjacent asphaltic concrete surface. Allow the specimen to cool to standard laboratory temperature, then trim any overfill flush, using a hot knife or spatula.

4.4.8.2 Procedure. Place the specimens in a forced-draft oven maintained at $60\pm3^{\circ}\text{C}$ ($140\pm5^{\circ}\text{F}$) for 168 ± 2 hr. Inspect at least once each day for specimen damage. Immediately after removing from the oven and again after cooling to standard laboratory temperature, examine the specimens for incompatibility of sealant with the asphaltic concrete. Determine conformance to the requirements of 3.8.

4.5 Toxicological Data and Formulations. The manufacturer shall provide a listing of the components in the sealant that when heated could produce hazardous vapors (see 5.3.3). Where precautions need to be taken relative to the inhaling of, or skin and eye contact with the material or vapors, these precautions shall be included in the manufacturer's instructions (see 3.9 and 5.3.3.1).

4.6 Inspection of Preparation for Delivery.

4.6.1 Sampling. Sampling for inspection of filled containers shall be in accordance with MIL-STD-105, inspection level II. The unit of product shall be one unit prepared for shipment.

4.6.2 Examination. Each filled container selected shall be inspected for conformance to the requirements of Section 5. Inspection shall be based on an Acceptable Quality level of 2.5 percent defective.

5. PREPARATION FOR DELIVERY

5.1 Packing. Packing shall be Level A, B, or Commercial as specified (see 6.2).

5.1.1 Level A. The material shall be packed in a close-fitting, tapered 24-gage metal pail with gasket and lug cover. Pails shall have a wire handle securely attached to ears or clips which shall be attached to the body of the

pails. The exterior surfaces of the pails shall be coated as specified in PPP-P-704. The unit pack quantity shall be one unit of issue quantity specified in the contract or purchase order.

5.1.2 Level B. The material shall be packed the same as for Level A except that the exterior surfaces of the pail shall be coated with a commercial coating.

5.1.3 Commercial. The material shall be packed to insure carrier acceptance and safe delivery at the destination in containers complying with the rules and regulations applicable to the mode of transportation.

5.2 Palletization.

5.2.1 Level A. Unless otherwise specified (see 6.2), material shall be palletized in accordance with MIL-STD-147.

5.2.2 Level B and Commercial. When specified (see 6.2), material shall be palletized in accordance with MIL-STD-147.

5.3 Marking.

5.3.1 Civil agencies. Shipment to civil agencies shall be marked in accordance with FED-STD-123.

5.3.2 Military agencies. Shipments to military agencies shall be marked in accordance with MIL-STD-129.

5.3.3 Special marking. In addition to the marking of 5.3.1 or 5.3.2, and any special marking of the contract or order, the following information will be shown on each pail:

- a. Name of sealant
- b. Specification number
- c. Manufacturer's name and material designation
- d. Manufacturer's lot and batch number

- e. Date of manufacturer (month and year)
- f. List of hazardous components (see 4.5)
- g. Quantity of sealant in pail (net weight)
- h. Application or pouring temperature
- i. Safe heating temperature
- j. Instructions for use

5.3.3.1 Instructions for Use. The instructions for use (see 6.8) shall include but not limited to the following: ambient temperature and humidity ranges, and moisture conditions of joints, for successful installation; essential requirements for preparation of joints, heating of the sealant, handling, placing, and disposal of the hot materials; and any restrictions to be adhered to in order to reduce hazards to personnel or to the environment. If it is not feasible to include all the instructions on the container without sacrificing legibility, the most important information shall be shown on the container and the full instructions referenced and furnished separately.

6. NOTES

6.1 Intended Use. This sealant is intended for sealing joints and cracks in pavements in rigid or flexible pavements that are not exposed to spillage of jet fuels and lubricating oils. It is not intended to be resistant to the heat and blast of jet aircraft engines, except when aircraft are moving at moderate speeds.

6.2 Ordering Data. Purchasers shall select the preferred options permitted herein, and include the following in procurement documents:

- a. Title, number and date of this specification
- b. When stability samples are required, quantity to be retained and by what activity (see 3.8 and 6.4)
- c. Addresses for submission of MSDS (see 3.10 and 6.5)
- d. Sampling, if other than as specified (see 4.3)

- e. Designation of Government approved test facility (see 4.4)
- f. Density and asphalt content values for asphaltic concrete pavement mix design and design method required (see 4.4.7.1)
- g. Level of packing required (see 5.1)
- h. If palletization is not required for Level A (see 5.2.1)
- i. When palletization is required for Level B or Commercial (see 5.2.2).

6.3 Data Requirements. When this specification is used in an acquisition which incorporates a DD Form 1423, Contract Data Requirements List (CDRL) and invokes the provisions of paragraph 52.227-7031 of the Federal Acquisition Regulations (FAR), the data requirements will be developed as specified by an approved Data Item Description (DD Form 1664) and delivered in accordance with the approved CDRL (DD Form 1423) incorporated into the contract. When the provisions are not invoked, the data shall be delivered in accordance with the contract requirements (3.10).

6.4 Stability Samples. The date of delivery shall be marked on samples submitted for stability testing (see 3.8).

6.5 MSDS Submission and Forwarding. MSDS copies will be forwarded to the designated Industrial Hygienist and the focal point of the activity that purchased the item, and the focal point of the using activity if different from purchasing activity. After review and acceptance of MSDS by designated recipients, approved copies will be forwarded to arrive at the destinations prior to material delivery (see 3.10).

6.6 Availability of Testing Materials and Apparatus. Known suppliers of specified testing materials and apparatus are as follows:

6.6.1 Melting Unit. A unit as specified in 4.4.2.1: Laboratory Melter Model BLM-100, Berry Corporation, P.O Box 337, Nicholasville, KY 40356.

6.6.2 Release Agent. An agent as specified in 4.4.4.1: Dow Corning 20 release coating, Dow Corning Corporation, Midland, MI 48640.

6.6.3 Concrete Blocks. Blocks as specified in 4.4.6.2: US Army Corps of Engineers, Missouri River Division Laboratory, 420 South 18th Street, Omaha, NE 68102.

6.6.4 Blotting Paper. Paper as specified in 4.4.6.3: White Reliance Blotting Paper, Product Code 13-01-12, James River Paper Company, Incorporated, 145 James Way, Southampton, PA 18966.

6.7 Asphalt Compatibility Specimens. The specimens of hot-mix asphaltic concrete as specified in 4.4.7.1 may be prepared using the methods described in ASTM D1074, D1559, or D1561.

6.8 Precaution. The sealant material can be damaged by heating at too high a temperature, reheating, or by heating for too long a period of time. The temperature of the sealant in the melting equipment should never exceed the safe heating temperature set by the manufacturer. Any given quantity of material should never be heated at the application or pouring temperature for more than 3 hr and should never be reheated. Sealant left in the equipment at the end of the working day should be removed and discarded. The sealant should be heated in a kettle or tank constructed as a double boiler, with the space between the inner and outer shells filled with heat transfer oil. Positive thermostatic control, mechanical agitation, and recirculating pumps should be provided to maintain uniform temperature of the sealant and heating oil. Direct heating is not permitted. Thermometers should be provided for continuous temperature readings of both oil and the sealant.

APPENDIX D: SUGGESTED SPECIFICATION FOR PRIMER, JOINT, NON-JET-
FUEL-RESISTANT, COLD-APPLIED, SINGLE COMPONENT
FOR PORTLAND CEMENT CONCRETE PAVEMENTS

1. SCOPE

1.1 Scope. This specification covers one type of one-component, cold-applied material for use in priming joints and cracks in portland cement concrete pavements prior to sealing the joint.

2. APPLICABLE DOCUMENTS

2.1 The following documents form a part of this specification.

Federal Specification

SS-S-1401C - Sealant, Joint, Non-Jet-Fuel-Resistant, Hot-Applied, for Portland Cement and Asphalt Concrete Pavements

2.2 Other publications. The following documents form a part of this specification to the extent specified herein.

American Society for Testing and Materials (ASTM)

C711-72 - Low Temperature Flexibility and Tenacity of One-Part, Elastomeric, Solvent-Release Type Sealants

D5-83 - Penetration of Bituminous Materials

D2823-75 - Standard Specification for Asphalt Roof Coatings

2.3 Order of precedence. In the event of a conflict between the text of this specification and the references cited herein, the text of this specification shall take precedence.

3. REQUIREMENTS

3.1 Description.

3.1.1 Material. The primer shall be a single-component, liquid composition that cures after application and which can be spray or brush applied to pavement crack or joint surfaces at temperatures between 50 and 120°F. The manufacturer shall specify the specific sealant products with which the primer is compatible and approved for use.

3.1.2 Performance. The primer shall cure to form a well-bonded tough surface coating which is non-tacky. The cured primer coating shall be fully compatible with manufacturer approved sealant materials and shall in no way adversely affect sealant performance characteristics. The cured primer coating shall be capable of filling and sealing voids in portland cement concrete joint faces to assist in restricting passage of moisture.

3.2 Viscosity. Viscosity shall not be less than 25 cp, or shall not exceed 250 cp when the primer is tested as specified in 4.4.3.

3.3 Tack Free Time. Tack free time shall not exceed 10 min when the primer is tested as specified in 4.4.4.

3.4 Cured Tack Free Evaluation. The primer shall pass the cured tack free evaluation at 60 min when tested as specified in 4.4.5.

3.5 Non-Volatile Content. The non-volatile content shall not be less than 20 percent and shall not exceed 40 percent when the primer is tested as specified in 4.4.6.

3.6 Permeability. Permeability shall not exceed 10 percent when the primer is tested as specified in 4.4.7.

3.7 Low-Temperature Flexibility. None of the specimens shall develop any cracks greater than 1/4 in. in length or exhibit any adhesion loss when tested as specified in 4.4.8.

3.8 Adhesion. No specimen shall develop any crack, separation, or opening between the sealant and the concrete blocks when the primer is tested as specified in 4.4.9.

3.9 Storage Stability. When specified, the Government will retain samples for verification of these requirements: The primer, when stored for 2 years from the date of delivery, at temperatures from -18° to 46°C (0° to 115°F), and tested in accordance with this specification, shall meet all of the requirements herein.

3.10 Toxicity. The material shall have no adverse effect on the health of personnel when used for its intended purpose. Questions pertinent to this effect shall be referred by the acquiring activity to the appropriate medical service who will act as advisor to the acquiring activity. The manufacturer's instructions shall provide personnel protection to meet OSHA requirements, including 29 CFR 1910.1000, 1910.1002, and 1910.1017, as applicable (see 4.5).

3.11 Material Safety Data Sheets (MSDS). MSDS shall be prepared in accordance with FES-STD-313 and submitted as directed in the contract or order at the time of acquisition award.

4. QUALITY ASSURANCE PROVISIONS

4.1 Responsibility for Inspection. Unless otherwise specified in the contract or purchase order, the contractor is responsible for the performance of all inspection requirements as specified herein. Except as otherwise specified in the contract or purchase order, the contractor may use his own or any other facilities suitable for the performance of the inspection requirements specified herein, unless disapproved by the Government. The Government reserves the right to perform any of the inspections set forth in the specification where such inspections are deemed necessary to assure supplies and services conform to prescribed requirements.

4.1.1 Materials inspection. The contractor is responsible for insuring that supplies and materials are inspected for compliance with all the requirements specified herein and in applicable referenced documents.

4.2 Quality conformance inspection. The quality conformance inspection shall be as specified in 4.4. Sampling shall be in accordance with 4.3.

4.3 Sampling. Unless otherwise specified, samples for testing shall be taken at the point of manufacture in accordance with ASTM D140. It shall be the responsibility of the contractor to determine that the samples taken are

representative of the batches proposed for shipment. The representative composite sample of the primer shall consist of not less than 2 liters (0.52 gal) from each batch. A lot consisting of a single batch, if taken from filled containers, shall be sampled by withdrawing approximately equal portions from three containers, selected at random, to make up the composite sample. The sample shall immediately be placed in an appropriate airtight sealed container which is either metal or plastic, in order to prevent loss of volatiles prior to testing. Sample identification shall include the name of the testing agency and contract or purchase order number. Each container from which sample material has been taken shall be resealed and marked for identification.

4.4 Testing. Testing shall be conducted at a Government-approved facility. Samples taken as specified in 4.3 shall be tested as specified in 4.4.1 through 4.4.9. Individual test values, and results of failure analyses of individual specimens, shall be recorded. Failure of the primer to pass any test shall be cause for rejection of the lot, except as noted for a marginal test result, following: where results of only one of the specified tests are judged by the testing agency to be marginal as to meeting requirements, the testing agency has the option to perform a retest. If the testing agency does not exercise its option to retest, either the contractor or Government may request that a retest for that property be made at the requester's expense. Such a test will be made only when an adequate quantity of the original sample is available or where additional material can be obtained from the previously marked sampled containers.

4.4.1 Standard conditions. Laboratory atmospheric conditions, hereinafter referred to as standard conditions, shall be $23 \pm 2^{\circ}\text{C}$ ($73 \pm 4^{\circ}\text{F}$) temperature and 50 ± 5 percent relative humidity. Specimens shall be stored and tested at standard conditions unless otherwise specified.

4.4.2 Specimen preparation. Prior to beginning testing, the sample shall be stored at laboratory standard conditions for a minimum of 24 hr in order to temperature condition the sample prior to opening the container. Since the primer contains volatile constituents, care must be taken during handling to minimize volatile loss. Therefore, the sample container should remain tightly closed at all times except when required for testing purposes. Just prior to initiating all testing, the sample container shall be opened and the sample shall be stirred to assure uniformity.

4.4.3 Viscosity.

4.4.3.1 Specimen preparation. The test sample be prepared by pouring 700 ± 100 ml. of the primer into at standard 1 quart (0.946 l) metal open top can with double friction lid. The lid shall then be placed on the can and the can sealed. The container shall then be placed in a constant temperature water bath as specified in ASTM D5, which is maintained at $77 \pm 0.2^{\circ}\text{F}$ for 1.5 to 2 hr.

4.4.3.2 Test. Remove the can from the water bath and remove the lid. Stir the sample to assure uniformity. Test for viscosity using a Brookfield Model HAT viscometer using Probe No. 1 at a speed of 50 rpm. Determine conformance to the requirements of 3.2.

4.4.4 Tack Free Time.

4.4.4.1 Specimen Preparation. Obtain one 2" x 3" x 1" concrete test block prepared in accordance with sections 4.4.6.2.1 and 4.4.6.2.2 of FS SS-S-1401C. Remove the blocks from the storage water and scrub the 2 by 3 in. faces with a stiff bristle brush under running water. Then lightly blot the surfaces of the block with an oil-free, soft absorbent cloth or paper to remove all free surface water. Place the block on a 2" x 1" face and permit to dry at standard conditions for 60 min.

4.4.4.2 Block Coating. Pour approximately 700 ml of primer into a standard 1 quart (0.946 l) open top metal can. Immerse the prepared and dried concrete block totally into the primer for 30 sec. Remove the block from the primer using forceps which grip the 1" x 3" sides, and hold the block with the 3 in. sides in a vertical position over the primer container and allow to drain for 60 sec.

4.4.4.3 Testing. Immediately after the draining period, place the coated block with the 2" x 3" side down on any appropriate level surface at laboratory standard conditions of $23 \pm 2^{\circ}\text{C}$ ($73 \pm 4^{\circ}$) and 50 ± 5 percent relative humidity for 10 min. Place a 150 by 25 by 0.1 ± 0.025 mm (6 by 1 by 0.004 ± 0.001 in.) polyethylene film on top of the primer coated block surface with the 6 in. axis of the film aligned with the 3 in. axis of the block. Load the polyethylene film for 30 sec with a metal plate approximately 41 by 29 mm (1.6 by 1.1 in.), with not less than 30 or more than 31 grams (g) mass. Remove the plate, and withdraw the film, uniformly and progressively, at right angles to the surface of the primer. Primer adhering to the polyethylene film shall constitute failure to conform to the requirements specified in 3.3.

4.4.5 Cured Tack Free Evaluation.

4.4.5.1 Specimen preparation. Prepare one block in accordance with section 4.4.4.1 of this specification.

4.4.5.2 Block Coating. Coat the block in accordance with section 4.4.4.2 of this specification.

4.4.5.3 Testing. Immediately after the draining period, place the coated block with a 2" x 3" side down on an appropriate level surface at laboratory standard conditions for 60 min. Place a 150 by 25 by 0.1 ± 0.025 mm (6 by 1 by 0.004 ± 0.001 in.) polyethylene film on top of the primer coated block surface with the 6 in. axis of the film aligned with the 3 in. axis of the block. Load the polyethylene film at the center of the block with a metal plate approximately 41 by 29 mm (1.6 by 1.1 in.) with not less than 30 or more than 31 grams (g) mass. Place the block, film, and weight in a forced draft oven maintained at 158°F for 60 min. Remove the specimen from the oven and immediately. Remove the plate, and withdraw the film, uniformly and progressively, at right angles to the surface of the primer. Primer adhering to the polyethylene film shall constitute failure to conform to the requirements specified in 3.4.

4.4.6 Non-Volatile Content.

4.4.6.1 Procedure. Test according to the Non-Volatile Matter determination of ASTM D2823-75 section 8.2. Determine non-volatile matter content and determine conformance to the requirement of 3.5.

4.4.7 Permeability.

4.4.7.1 Specimen Preparation. Prepare three test blocks in accordance with section 4.4.4.1 of this specification except instead of allowing to air dry for 60 min, determine and record the saturated surface-dried weight of each block (A) to the nearest 0.01 gram, immediately after blotting to remove free surface water. Then, dry the blocks to constant weight at $135 \pm 5^{\circ}\text{C}$ ($275 \pm 9^{\circ}\text{F}$). Constant weight shall be verified by successive weighing using a scale accurate to 0.01 gm. Dry until the blocks show a weight change of not more than 0.05 gm on successive hourly weighing. Each weighing should be after cooling in a desiccator. Label each block and record the oven-dried weight (B) to the nearest 0.01 gm. Store dried blocks in a desiccator until they are used.

4.4.7.2 Block Coating. Coat each block with primer following the procedure in section 4.4.4.2 of this specification. After the 60 sec draining period, support the block with one of the 2 in. by 3 in. sides down on a fixture with four vertical supporting pins, as shown in Figure 1. Cure the supported block for 4 hr at standard conditions.

4.4.7.3 Test Procedures. Weigh the block and primer to the nearest 0.01 gm at the end of the 4 hr curing period and record the weight as C. Place each block in a 1 liter glass beaker which contains 500 ± 50 ml of distilled water which is at $25 \pm 1^{\circ}\text{C}$ ($77 \pm 2^{\circ}\text{F}$). Loosely cover each beaker and place in an oven or water bath to maintain the $25 \pm 2^{\circ}\text{C}$ temperature for 60 min. At the end of the 60 min period, remove the blocks from the water, blot the surfaces with an oil free soft absorbent cloth, to remove all free surface moisture. Immediately weigh to the nearest 0.01 gm, and record the weight as D.

4.4.7.4 Calculations. Calculate permeability as the percentage of weight gained during the 60 min soaking period at 25°C as a percentage of the saturated surface dried moisture content of the block as follows:

A - Saturated surface dried weight of uncoated block

B - Oven-dried block weight

C - Block weight when coated with primer

D - Saturated surface dried weight of soaked coated block

$$\text{Permeability} = \frac{D - C}{A - B} \times 100 \text{ percent}$$

Calculate the permeability for each of the three blocks average the results, and then determine conformance to the requirements specified in 3.6.

4.4.8 Low Temperature Flexibility.

4.4.8.1 Specimen Preparation. Test specimens are prepared in accordance with the ASTM C711-72 procedure. Due to the low viscosity of the primer, it will be necessary to seal the interface between the template and the plate with a solvent resistant sealer (such as Pearmatex No. 2, Pliable, Non-Hardening Gasket Sealant supplied by Loctite Corporation, Automotive and Consumer Group, Kansas City, Kansas 66115) to prevent leakage of the primer. The sealed template and plate shall then be filled with primer and conditioned at lab standard conditions for 24 ± 2 hr. Remove the template after conditioning with the aid of a thin knife by cutting around the outside edge of the primer.

4.4.8.2 Testing Procedure. Expose the conditioned specimen to $158 \pm 3.6^\circ\text{F}$ ($70 \pm 2^\circ\text{C}$) for 16 hr, and then further condition the specimen at $32 \pm 1^\circ\text{F}$ ($0 \pm 0.5^\circ\text{C}$) for 1 hr. After the 1 hr conditioning at 32°F , bend the panel through 180° over the $1/4$ in. diameter mandrel with the primer side uppermost. The bend shall be performed in not less than 1 s and not more than 1.5 s. Immediately after bending examine the primer for cracking, separation, delamination, and adhesion loss. Minor surface crazing or hairline cracks and minor edge cracking may be ignored. Determine conformance to requirements of section 3.7.

4.4.9 Adhesion.

4.4.9.1 Specimen Preparation. Prepare 6 concrete blocks as specified in sections 4.4.4.1 and 4.4.4.2. Then, place a 2 in. by 3 in. surface of the coated block on an appropriate level surface and allow to cure at laboratory standard conditions for 60 min. Assemble three sets of blocks in preparation for pouring sealant in accordance with section 4.4.6.3 of FS SS-S-1401C. Heat sealant recommended by the manufacturer which meets FS SS-S-1401C in accordance with section 4.4.2 and prepare specimens using the primer coated blocks in accordance with section 4.4.6.3 of FS SS-S-1401C.

4.4.9.2 Testing. Test the prepared samples for nonimmersed and water-immersed bond using procedures specified in sections 4.4.6.4 and 4.4.6.5 of FS SS-S-1401C. Examine the results as specified in section 4.4.6.6 of FS SS-S-1401C. Determine conformance to the requirements of section 3.8 of this specification.

4.5 Toxicological data and formulations. The manufacturer shall provide a listing of the components in the primer that could give off hazardous vapors during use. Where precautions need to be taken relative to the inhaling of, or skin and eye contact with the vapors, these precautions shall be included in the manufacturer's instructions (see 3.10).

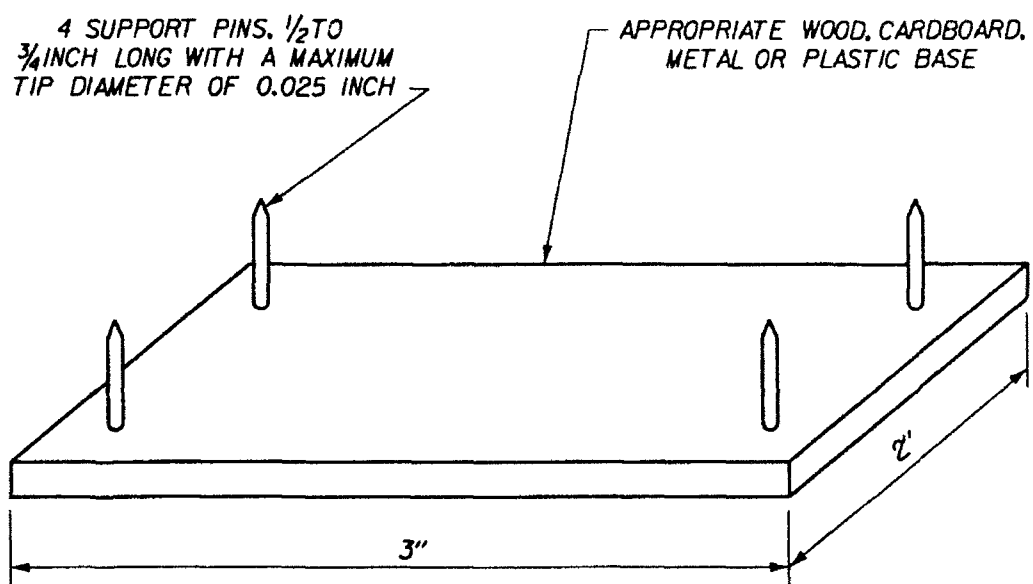


Figure D1. Primer coated block support fixture for drying of coated concrete blocks

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13. ABSTRACT (Maximum 200 words) A joint effort research program between Crafcro Incorporated and the US Army Engineer Waterways Experiment Station was conducted under the auspices of the FY 89 Construction Productivity Advancement Research Program. The purpose was to develop improved materials and processes for sealing and resealing joints in portland cement concrete pavements. The program's objectives were to (a) develop specification limits for a hot-applied, jet-fuel-resistant (JFR) sealant with improved characteristics as compared to current Federal Specification (FS) SS-S-1614A requirements, (b) develop specification limits for a hot-applied, non-jet-fuel-resistant (non-JFR) sealant with improved low temperature performance characteristics as compared to current FS SS-S-1401C requirements, (c) develop specification limits for a primer system that will minimize bubbling tendencies associated with hot-applied sealants and improve adhesion to portland cement concrete, and (d) develop field data to determine performance characteristics of				
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flush fill sealant application geometry versus 1/8 to 1/4 in. recess application techniques. This report summarizes the laboratory investigation phases of the research program. Field evaluations of the materials developed to meet these specifications are being conducted to determine actual maintenance cost reductions and improved construction quality.

Prior to initiating sealant development activities, testing of available sealant materials was performed to identify the level of laboratory performance of currently available hot-applied, JFR and non-JFR sealants. Thirteen different sealants from four manufacturers were tested for specification conformance to FS SS-S-1614A and FS SS-S-1401C. Additional testing procedures were also used to aid in determining performance limits. Testing verified that the JFR sealants tested were not capable of passing bond tests at -20°F, and that they experience significant amounts of surface hardening when subjected to 158°F oven-aging. Non-JFR sealants tested varied in low temperature extension capabilities. Products which met low-modulus ASTM D 3405 specifications were capable of passing bond testing at -20°F using 200 percent extension, while the FS SS-S-1401C materials could not.

Laboratory development work determined that it was possible to produce hot-applied, JFR sealant materials which could pass bond testing at -20°F, and with less surface hardening after aging. A suggested specification was developed for this improved material, and a pilot production batch of sealant meeting this specification was made. Additionally, a suggested specification for a low-modulus non-JFR sealant with improved low temperature properties was developed, and a pilot production batch of sealant meeting this specification was made. Both sealants were installed at Fairchild AFB, WA, for field evaluation. The results of the field evaluation of the improved sealants versus commercially available sealants are presented in a separate report.

Bubbling characteristics of sealants were studied using the US Army Corps of Engineers' Handbook for Concrete and Cement CRD-C 525-89 procedure along with several modifications to better understand the bubbling problem. Data indicate that as little as 0.5 percent moisture in portland cement concrete (PCC) can cause sealant bubbling. A quantitative method to monitor bubbling rate was developed. A variety of potential encapsulating primer formulations were studied. Results indicated that when the appropriate primer system is used on PCC, swelling and bubbling of sealants can be reduced. Appropriate adhesion could not be obtained between the sealant and PCC pavement when the developmental JFR primers were used. The developmental non-JFR primers increased sealant adhesion and reduced sealant bubbling in the laboratory. A suggested specification for primer for use with non-JFR sealant for PCC pavements was developed, and a pilot production batch of primer to meet this specification was made for field evaluation at Fairchild AFB, WA.